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REVIEW OF THE DEPARTMENT OF THE ARMY PESTICIDE MONITORING PROGR--ETC(U)
MAR 81 K L OLDS, J H VINOPAL, J F SUPROCK

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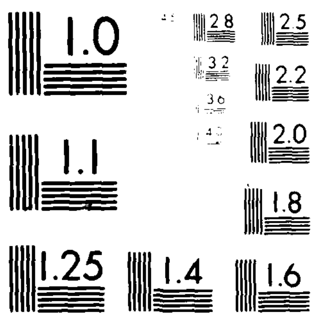

MICROCOPY RESOLUTION TEST CHART

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UNITED STATES ARMY ENVIRONMENTAL HYGIENE AGENCY

ABERDEEN PROVING GROUND, MD 21010

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PART II
PESTICIDE MONITORING SPECIAL STUDY NO. 17-44-0230-81
REVIEW OF THE DEPARTMENT OF THE ARMY
PESTICIDE MONITORING PROGRAM:
EVALUATION OF SOIL AND SEDIMENT SAMPLES
COLLECTED DURING CALENDAR YEARS 1975-1978.

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REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER 17-44-0230-81	2. GOVT ACCESSION NO. AD-A098286	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Part II. Pesticide Monitoring Special Study No. 17-44-0230-81. Review of the Department of the Army Pesticide Monitoring Program: Evaluation of Soil and Sediment Samples Collected During CY 1975-1978		5. TYPE OF REPORT & PERIOD COVERED Review Report 1975-1978
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) KENNETH L. OLDS J. HOWARD VINOPAL, Ph.D., R.P.E. JOHN F. SUPROCK THOMAS M. WHITE		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS US Army Environmental Hygiene Agency Aberdeen Proving Ground, MD 21010		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS Commander US Army Health Services Command Ft Sam Houston, TX 78234		12. REPORT DATE March 1981
		13. NUMBER OF PAGES 56
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) UNCLASSIFIED
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

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17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Pesticides	Residues
Monitoring	Analysis
Environment	Pesticide Management
Soil	
Sediment	

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Results of four years monitoring data for soil and sediment collected as part of the Department of the Army Pesticide Monitoring Program are presented. Significant findings are as follows:

a. The various land use stratifications show significant ($p=0.05$) differences in the mean amount of pesticide residues present. The land use stratification having the highest pesticide concentrations are shop and storage areas followed by golf courses, sewage treatment and landfill, and residential. Sediment samples consistently have the lowest concentrations.

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b. Four years data show no significant temporal trends with the exception of stream exits which show a significant upward trend.

c. Comparison of DAPMP data with other published data show comparable soil residue levels. Army sediment residues are comparable to those found in the San Francisco Bay, but are higher than those obtained in a Canadian study.

d. The four years data provide the basis for the formation of quality control charts. These charts allow the identification of potential problem areas and a comparison of a one time installation sampling to the DAPMP data base.

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Mr. Olds/clf/AUTOVON
584-3613

REPLY TO
ATTENTION OF
HSE-RP/WP

23 MAR 1981

SUBJECT: Part II, Pesticide Monitoring Special Study No. 17-44-0230-81, Review of the Department of the Army Pesticide Monitoring Program, Evaluation of Soil and Sediment Samples Collected During Calendar Years 1975-1978

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Results of the Department of the Army Pesticide Monitoring Program (DAPMP) Evaluation of Soil and Sediment Samples Collected during CY 75-78 are presented in the inclosed report. Significant findings and recommendations are as follows:

a. Findings. The various land use stratifications show significant differences in the mean amount of pesticide residues present. Data from four successive collection years utilizing the same sampling scheme show no significant trend, either upward or downward, with the exception of stream exits which show a significant upward trend. Comparisons of DAPMP data with other published data show comparable residue levels. The four years' data provide the basis of quality control charts which aid in the identification of possible or potential problem areas. These charts also provide a means by which a one-time monitoring of an installation can be compared with the DAPMP data base.

b. Recommendations.

(1) Future sampling of DAPMP installations should be redirected to air and suspendible dust samples from those areas of intense human occupancy. Efforts should be made to complete analysis of samples on hand. Increased efforts should be directed toward the development of analytical methodology for those pesticides having the most frequent use in the Army.

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(2) The quality control charts should be used to evaluate a one-time sampling of an installation to determine possible problem areas.

FOR THE COMMANDER:

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U. S. ARMY ENVIRONMENTAL HYGIENE AGENCY
ABERDEEN PROVING GROUND, MARYLAND 21010

REPLY TO ATTENTION OF

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PART II
PESTICIDE MONITORING SPECIAL STUDY NO. 17-44-0230-81
REVIEW OF THE DEPARTMENT OF THE ARMY PESTICIDE MONITORING PROGRAM: EVALUATION OF SOIL AND SEDIMENT SAMPLES COLLECTED DURING CALENDAR YEARS 1975-1978

1. AUTHORITY. Letter, DASG-PSP-E, Office of The Surgeon General, 9 October 1979, subject: AMEDD Pest Management Program.

2. REFERENCES. See Appendix A for a listing of references.

3. PURPOSE. Data collected over a 4-year period have a high probability of indicating the following, which are evaluated in this report:

a. Adequacy of the sample of installations.

b. Adequacy of the environmental sampling plans and their execution.

c. Possible secular trends for the pesticides analyzed for.

d. The existence of specific land use area problems.

4. BACKGROUND.

a. Stratified samples of soil and sediment have been collected from a sample of Army installations over a period of 4 years. Random fish and bird samples have also been collected from certain of these installations.

(1) Reference 1, Appendix A, indicates the number of installations comprising the sample of Army installations. In particular, this reference indicates the difficulty of obtaining complete fish and bird sampling.

(2) References 3 and 5, Appendix A, indicate a close association between the fish and sediment samples and between the soil and bird samples. These active biological components, i.e., the fish and birds, did indicate a greater diversity of pesticides, but this diversity is the result of pesticide metabolities rather than pesticides per se.

(3) In order to expedite this report, and in view of the information cited above, only the results of soil and sediment analyses are evaluated in this part. Data from the fish and bird samples collected over the 4-year period, to the extent collections were sufficiently complete, will be evaluated in Part III.

b. Additional compounds have been added to the analyses for the samples collected in 1977 and 1978 compared with all earlier samples. In particular, the polychlorinated compounds, polychlorinated biphenyls (PCBs), and several chlorophenoxy herbicides (2,4-D, 2,4,5-T, etc.) are being added. The PCBs were recommended as a part of pesticide monitoring by Schecter (1971). (See Appendix E, Literature Cited.) When present in environmental samples, the PCBs give rise to a complex series of peaks on gas chromatograms, and some of the peaks overlap or coincide with those given by some of the common organochlorine pesticides. The PCBs are relatively persistent toxic chemicals and, because of their adverse ecological effects, should be reported whenever detected in monitoring samples. Unfortunately, the chlorophenoxy herbicides are not included in the classical multiple residue methods. Inclusion of these pesticides in routine monitoring more than doubles the analytical workload.

c. The detection of secular trends, to the extent of their existence, as mandated by Congress (PL 92-516) requires particular and specific attention to the faithful execution of a tested sampling plan. In view of the absolute limitations imposed by the sample, it is imperative that all operations--sampling, analyses, evaluation and interpretation--be fully integrated and coordinated. Unfortunately, this has not been possible in all cases during the 4 years of sampling evaluated in this report.

d. Assurances of data reliability, in addition to the classical constraints imposed by the sample, also require the commitment of a significant portion of available resources to intralaboratory and interlaboratory quality control programs. A description of the Department of the Army Pesticide Monitoring Program (DAPMP) quality control program is given in Appendix B.

e. The statistical and chemical analysis procedures were described in reference 3, Appendix A. To provide emphasis on the importance of transformed data, the untransformed means are occasionally tabulated.

5. RESULTS AND DISCUSSION. The data presented utilize the data transformation described in reference 3, Appendix A. The data are evaluated on the basis of selected chemical classes and subgroups. These are described in Appendix C. Comparisons are made on the land use stratifications described in references 4 and 5, Appendix A.

a. Figures 1-20 (Appendix D) show comparisons of the various land use areas for the 4-year period. The diversity indices in Figures 5 and 6 are computed from the following formula, $H' = \Sigma_i p_i \log p_i$, where p_i is the proportion of the pesticide in the sample to the total concentration for that pesticide. The higher the diversity index the more uniform the distribution of the pesticides. Figures 7-10 and Figures 15-18 show no values for 1976. This is the result of analysis of only selected samples for that year. Figures 1-20 demonstrate that the various land use areas are significantly different ($P = 0.05$). For the soil groups there are no significant temporal trends in the data over the 4-year period. The only well defined and statistically significant pattern in the sediment is found in stream exits. The increase observed from 1976 to 1978 is the result of more compounds being observed and a more uniform distribution of the pesticides being found. The increase is attributable to increased DDT residues and increased cyclodiene residues. However, since there are only 3 to 4 years' data on which these yearly trends are based, additional data are needed to determine actual trend patterns.

b. Of particular interest in the soil data, Figures 7-10, is the lack of appreciable degradation of DDT residues during the 4-year period. The persistence of chlorinated hydrocarbon pesticides in soil is very dependent on soil type, climatic conditions and microorganism populations (Edwards, 1970). The breakdown of chlorinated hydrocarbons in the soil is not truly exponential; however, half-lives can be estimated for any pesticide (Edwards, 1970). Reported half-lives for DDT range from approximately 8 months to 6 years (Fleming and Maines, 1953) (Lichtenstein and Shulz, 1959) (Gambrell et al, 1968). These half-lives are very dependent upon the original concentration applied. The pattern of degradation appears to approximate a sigmoid curve when remaining concentrations are plotted against time, Figure 21 (Appendix D). As DDT was banned from use in 1972, the residues observed may be those remaining after the first initial half-life disappearance. Gambrell (1968) suggests that the second half-life for DDT may be in the range of 2 to 5.5 years. Based on the variations reported in the first half-life, the actual length of time for the second half-life could easily exceed the 5.5 years reported. As a result, many years of monitoring may be required to establish an overall decreasing trend in the DDT residues. The reported half-lives of the other persistent chemicals are only slightly shorter than those reported for DDT (Edwards, 1970). Thus, the absence of trends in Figures 12 and 13 are probably the result of factors similar to those affecting DDT degradation. The general absence of organophosphate residues, Figure 14, and the lack of any yearly trends are attributable to

two factors. The first is the rapid degradation of these compounds and the second is the possible degradation prior to extraction due to prolonged storage of CY 75-77 samples. Edwards (1972) reported that the most persistent organophosphates have a half-life of about 24 weeks with the majority being 6 weeks or less. Consequently, our finding of any organophosphate residues is probably the result of sampling shortly after an application or, in the case of shop and storage areas, sampling at the site of a massive or recent spill or around leaking containers.

c. Comparison of residue data from the DAPMP with monitoring data from other agencies and organizations is constrained by the lack of uniformity in sampling protocols and data transformation techniques.

(1) Table 1 presents a comparison of arithmetic mean residue values for soil collected from five United States cities by the US Environmental Protection Agency in 1971 (Carey, et al, 1979) compared with DAPMP and Air Force data. The DAPMP data include a consolidation of residential, cantonment and other recreation areas to more closely approximate the sampling described by Carey. Although comparisons utilizing arithmetic means are hindered by the wide range of residues, the values for the two programs appear to be quite comparable. Data collected by the Air Force (Lang, et al, 1979) also appear to be comparable to the DAPMP data with the exception of higher Air Force chlordane residues in 1975. Lang explains this high mean value as being attributable to Wright-Patterson Air Force Base where chlordane problems have been previously reported. Table 2 presents a comparison of the arithmetic mean data for pesticides found on golf courses on Army and Air Force installations. The higher means observed for the DAPMP data are probably the result of different sampling protocols. Lang, et al (1979) describes samples from Air Force golf courses as being "collected from random starting points at 45-ft (13.7-m) intervals along both sides of the fairway at the edge of the rough." The Army golf course samples were selected randomly, with all areas of the course having an equal chance of being chosen. If a particular sampling site was on a green, the location was moved to an area just adjacent to the green. Since the greens probably receive greater pesticide treatment than the fairways, this may account for the increased residues in the DAPMP data.

(2) Arithmetic mean data (untransformed) for DAPMP sediment samples are presented in Table 3. In previous work only 3 of 29 sediment samples collected from Aransas Bay in Texas were positive for pesticides (Fay and Newland, 1972). One of these samples contained 0.7-ppb dieldrin and another 0.5-ppb dieldrin. The third sample contained 24.6 ppb of p,p'-DDD. Law and Goerlitz (1974) reported their findings on 39 streams tributary to San Francisco Bay. A summary of their results is shown in Table 4.

TABLE 1. COMPARISON OF PESTICIDE RESIDUE LEVELS IN SOIL SAMPLES COLLECTED IN RESIDENTIAL AREAS AS PART OF MONITORING PROGRAMS CONDUCTED BY THE ARMY, AIR FORCE, AND THE US ENVIRONMENTAL PROTECTION AGENCY (EPA)

		Pesticide Residues Levels (ppm)														
Sample Designation	No. Samples	Aldrin	Dieldrin	Chlordane	Heptachlor	Heptachlor Epoxide	Endrin	Toxaphene	PCBs	o,p'-DDE	p,p'-DDE	o,p'-DDD	p,p'-DDD	o,p'-DDT	p,p'-DDT	ΣDDT
							Army*									
CY 75	297	0.003	0.249	0.371	0.001	0.012	0.001	0.005	---	0.002	0.270	0.014	0.047	0.106	0.621	1.061
76	126	0.007	0.409	0.266	<0.001	0.008	0.005	0	0.037	---	---	---	---	---	---	---
77	69	0.051	0.046	0.533	0	<0.001	<0.001	0	0.053	0.001	0.166	0.004	0.020	0.057	0.245	0.494
78	72	<0.001	0.015	0.038	<0.001	0.024	0	0	0.019	0	0.172	0.009	0.014	0.087	0.349	0.631
							EPA									
City #1	156	0.02	0.02	0.21	0	<0.01	0	0	0.02	<0.01	0.12	ND	0.13	0.02	0.14	0.40
2	55	0	<0.01	0.07	0	<0.01	0	0	0.22	0	0.04	ND	0.02	0.01	0.06	0.12
3	48	<0.01	0.06	4.00	<0.01	0.02	0	0	0	<0.01	0.17	ND	0.13	0.04	0.20	0.56
4	43	0	0.20	0.08	<0.01	<0.01	<0.01	0.24	0	<0.01	0.10	ND	0.01	0.05	0.20	0.37
5	78	0	0.04	0.16	<0.01	<0.01	0	0	0.04	0	0.11	ND	0.06	<0.01	0.08	0.26
							Air Force									
CY 75			0.01	5.43	0.01	<0.01	<0.01									0.86
76			<0.01	0.16	0	<0.01	0									0.63

* Army residential includes residential, cantonment, and other recreation sites.
- Not analyzed
Blank - Not reported
ND - None Detected

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TABLE 2. COMPARISON OF PESTICIDE RESIDUE LEVELS IN SOIL AND GOLF COURSES OF MONITORED ARMY INSTALLATIONS AND AIR FORCE BASES

Sample Year	No. Samples	Pesticide Residue Levels (ppm)														
		Aldrin	Dieldrin	Chlordane	Heptachlor	Heptachlor Epoxide	Endrin	Toxaphene	PCBs	o,p'-DDE	p,p'-DDE	o,p'-DDD	p,p'-DDD	o,p'-DDT	p,p'-DDT	ΣDDT
Army																
CY 75	88	0	0.116	2.317	0	0.028	0	0	---	0.03	22.30	0.56	1.35	8.62	35.69	0.78
76	33	0	0.513	0.182	0	0.002	0.005	0.07	0	---	---	---	---	---	---	---
77	32	0	0.031	0.326	0	0.001	0	0	0	0	5.93	1.55	1.44	5.46	33.72	1.503
78	33	0	0.385	2.671	0	0.005	0.001	0.212	0	0.08	14.72	0.08	0.36	3.87	20.74	1.208
Air Force																
CY 75	17		0.01	0.67	0	<0.01	0		0.18							0.19
76	17		0.01	0.56	0	<0.01	<0.01		0.24							0.16

- Not analyzed
Blank - Not reported

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TABLE 3. PESTICIDE RESIDUE LEVELS IN SEDIMENT SAMPLES COLLECTED ON ARMY INSTALLATIONS DURING THE PERIOD 1975-1978

			Pesticide Residue Levels (ppm)								
Sample Year	No. Samples		o,p'-DDD	p,p'-DDD	o,p'-DDE	p,p'-DDE	o,p'-DDT	p,p'-DDT	Chlordane	Dieldrin	PCBs
CY 75	363	$\overline{X}$	0.057	0.226	0.005	0.030	0.004	0.054	0.012	0.001	---
		Low	0	0	0	0	0	0	0	0	---
		High	18.25	67.08	1.71	6.71	0.76	8.34	1.62	0.10	---
		% Positive	7	16	2	14	2	5	4	2	---
CY 76	122	$\overline{X}$	---	---	---	---	---	---	0.003	0.001	0.002
		Low	---	---	---	---	---	---	0	0	0
		High	---	---	---	---	---	---	0.12	0.07	0.21
		% Positive	---	---	---	---	---	---	3.3	4.1	0.8
CY 77	107	$\overline{X}$	0.005	0.020	0.003	0.019	<0.001	0.002	0.004	<0.001	0.006
		Low	0	0	0	0	0	0	0	0	0
		High	0.18	0.73	0.12	0.86	0.02	0.08	0.14	0.013	0.68
		% Positive	6	11	4	8	2	3	4	2	1
CY 78	128	$\overline{X}$	0.018	0.078	<0.001	0.013	<0.001	0.016	0.002	0.002	0.052
		Low	0	0	0	0	0	0	0	0	0
		High	0.94	3.81	0.03	0.59	0.04	1.54	0.16	0.27	4.22
		% Positive	6	14	1	9	1	5	2	1	2

- Not analyzed

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TABLE 4. PESTICIDE RESIDUES (ppm) FROM 39 STREAMS TRIBUTARY TO SAN FRANCISCO BAY

Range	DDD	DDE	o,p'-DDT	p,p'-DDT	Chlordane	PCBs
$\bar{X}$	0.022	0.012	0.007	0.023	0.120	0.104
Low	0	0	0	<0.001	0	0
High	0.160	0.061	0.089	0.200	0.800	1.400

Klassen and Kadoum (1975), while studying the Tuttle Creek Reservoir in Kansas, reported no pesticide in any of the bottom sediments analyzed (limits of detectability = 0.01 ppm). In Ontario, Canada, Miles and Harris (1973) studied three bodies of water, one draining an agricultural area, one an urban-agricultural area, and another a resort area. In the agricultural area, DDT had been used on tobacco crops. The urban-agricultural area contained the city of London, Ontario, which drains 1200 square miles of mixed agricultural land (chiefly dairy cattle). The resort area studied had been subjected to DDT in previous years for biting fly control. Results of their assays are summarized in Table 5.

TABLE 5. PESTICIDE RESIDUES (ppm) IN SEDIMENT COLLECTED IN ONTARIO, CANADA

Range	DDT	p,p'-DDE	o,p'-DDT	p,p'-DDT	o,p'-DDD	p,p'-DDD	Dieldrin	Chlordane
Agricultural Area (collected Apr-Oct 71)								
$\bar{X}$	0.018	0.004	<0.001	0.006	<0.001	0.006	0.001	<0.001
Low	0.014	0.003	<0.001	0.004	<0.001	0.005	<0.001	<0.001
High	0.022	0.005	0.002	0.010	0.001	0.007	0.005	0.003
Urban-Agricultural (Collected Apr-Oct 71)								
$\bar{X}$	0.003	<0.001	-	<0.001	-	0.002	<0.001	-
Low	0.002	<0.001	-	0	-	0.001	<0.001	-
High	0.004	0.001	-	<0.001	-	0.003	<0.001	-
Resort (Collected May-Sep 71)								
$\bar{X}$	0.015	0.007	<0.001	0.002	<0.001	0.005	<0.001	<0.001
Low	0.009	0.004	<0.001	<0.001	<0.001	0.003	<0.001	<0.001
High	0.022	0.009	<0.001	0.004	0.001	0.007	0.001	0.002

The arithmetic mean data for sediment from the DAPMP resemble very closely the data of Law and Goerlitz (1974) for streams tributary to San Francisco Bay with the exception of the PCBs. The PCB data for the DAPMP do have a much higher maximum value than the San Francisco Bay data (4.22 ppm and 1.40 ppm, respectively), but the mean values for the DAPMP range from 2 to 50 times lower. This may be attributable to the sample size differences. However, both the DAPMP data and the San Francisco Bay data are higher for DDT and metabolites than the Canadian data of Miles and Harris. There is no ready explanation of this difference, particularly in light of the known usage of DDT in the Canadian areas.

d. Utilizing the transformed data obtained for the 4 years, an estimate can be made as to the "normal" pesticide residue level for an Army installation.

(1) From these data appropriate control charts can be determined using 95-percent confidence intervals for the various land use areas. These control charts provide a "ruler" for determining if a particular installation is or is not within the bounds of what one would expect at a "normal" installation. Figures 22 to 31 (Appendix D) present the control charts for the various land use areas for the 4 years' data. These data utilize transformed means for data from which DDT residues have been excluded as well as data including DDT. These figures show some outliers beyond the 95-percent confidence intervals. These outliers themselves indicate a possible problem at a given installation. When there appears to be a trend in the outliers, as in the case of installation six for the sediment sampling, this points toward a potential problem. A closer look at the 4 years' data for installation six shows that one river traversing the installation is responsible for the increase observed in 1977 and 1978. The data for the 4 years do not reveal a consistent pattern, but do reveal a need for further sampling to delineate the problem area.

(2) Although the control charts are useful in identifying existing or potential problem areas on the currently monitored installations, their use will be most beneficial for future, one-time monitoring of other installations. A one-time monitoring of an installation can then be compared to the control charts to determine possible problem areas. These control charts do not address the issue of what is an acceptable level of pesticides in a given land use area. At present there are no values established for acceptable levels of pesticides in these environments. The control charts present only an indication of what is "normal" for an Army installation. If other Army land use areas are comparable, as are the residential areas to other urban areas, an assumption can be made that Army installations are comparable to the general environment in the United States. The control charts then provide a "ruler" for an installation's standing with its neighboring environment.

6. CONCLUSIONS.

a. The various land use stratifications show significant (P = 0.05) differences in the mean amount of pesticide residues present. This is in agreement with results previously obtained (references 2 and 3, Appendix A). The land use stratifications having the highest pesticide concentrations are shop and storage areas followed by golf courses, sewage treatment and landfill, and residential. Sediment samples consistently have the lowest concentrations. A further breakdown of the sediment data reveals that the entrance and exit points of streams traversing installations are significantly higher than those streams originating on the installations and impounded bodies of water.

b. Data from four successive collection years utilizing the same sampling scheme show no significant temporal trend, either upward or downward, with the exception of the stream exits which show a signficant upward trend. The stream exits, although showing an upward trend, must be viewed in light of the other sediment data which show extreme variability from year to year. To fully evaluate this upward trend, additional data are needed.

c. Comparison of DAPMP data with other published data is constrained by different sampling plans and different data transformation techniques. A comparison of arithmetic means of DAPMP residential sampling with the National Urban Monitoring Program and Air Force data shows comparable soil residue levels. A similar comparison of Army golf course data with the Air Force data shows the Army having higher residues, probably the result of differences in sampling techniques. Pesticide residues in sediment from Army installations are similar to those found in a study of the San Francisco Bay, but are higher than those obtained in a Canadian study. This difference is unexplainable.

d. The 4 years' data provide the basis for the formation of quality control charts. These charts enable the determination of "normal" pesticide residue levels for the land use areas. Use of the quality control charts aids in the identification of possible or potential problem areas. These charts also provide a means by which a one-time monitoring of an installation can be compared with the DAPMP data base.

7. RECOMMENDATIONS.

a. Based on the absence of significant temporal trends of the currently monitored pesticides, it is recommended that future sampling of DAPMP installations be redirected to air and suspendible dust samples from those areas of intense human occupancy. Efforts should be made to complete analysis of samples on hand, and increased efforts should be directed toward the development of analytical methodology for those pesticides having the most frequent use in the Army.

b. The quality control charts should be used to evaluate a one-time sampling of an installation to determine possible problem areas.

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APPENDIX A

REFERENCES OF USAEHA REPORTS

1. Part I, Pesticide Monitoring Special Study No. 17-44-0230-80, Review of the Department of the Army Pesticide Monitoring Program.

2. Pesticide Monitoring Annual Report No. 17-44-0140-79, Department of the Army Pesticide Monitoring Program Evaluation of Environmental Samples Collected in Calendar Year 1976, ADA067267.

3. Pesticide Monitoring Annual Report No. 44-0100-78, Department of the Army Pesticide Monitoring Program Evaluation of Environmental Samples Collected in Calendar Year 1975, ADA050880.

4. Pesticide Monitoring Study No. 44-0142-78, Pesticide Monitoring Guidelines, Department of the Army Pesticide Monitoring Program (effective 1 April 1978), ADA047777.

5. Pesticide Monitoring Special Study No. 44-111-76, Pesticide Monitoring Guidelines, Scheduled Monitoring (effective 1 April 1976), ADA029983.

6. Pesticide Monitoring Special Study No. 44-0102-77, Environmental Sampling in the Panama Canal Zone, 1 December 1976, ADA034765.

7. Pesticide Monitoring Special Study No. 44-0131-77, Pesticide Recovery Studies for Evaluation of Department of the Army Pesticide Monitoring Program Soil and Sediment Analysis Methodology. Part I. Determination of Pesticide and Polychlorinated Biphenyl Recoveries From Soil Extracted Immediately Following Fortification, October-December 1976, ADA035782.

8. Pesticide Monitoring Study No. 17-44-0921-79, Evaluation of Silicic Acid Column Pesticide/Polychlorinated Biphenyl Separation Procedure: Recovery and Elution Patterns of 24 Pesticides and Pesticide Metabolities and Two Polychlorinated Biphenyls, September 1977 - February 1979, ADA067149.

APPENDIX B

ANALYTICAL QUALITY CONTROL PROCEDURES FOR THE DEPARTMENT OF THE ARMY PESTICIDE MONITORING PROGRAM 1975-1979

1. GENERAL.

a. Use of Standardized, Validated, and Published Analytical Methodology. Where available and feasible, standardized and validated published analytical methodology was used in the DAPMP. Basic reference sources for DAPMP analytical methodology and detailed descriptions of DAPMP sample preparation, extraction, cleanup and analysis procedures were presented in Appendix D of the CY 75, DAPMP Annual Report (reference 3, Appendix A). In addition, reports describing detailed in-house evaluations of DAPMP soil and sediment methodology (reference 7, Appendix A) and silicic acid column pesticide/PCBs separation methodology (reference 8, Appendix A) have been prepared.

b. Shipment and Storage of Samples. Shipment and storage procedures for DAPMP soil, sediment, fish, and bird samples were also discussed in Appendix D of the above cited DAPMP report (reference 3, Appendix A). Beginning with CY 78 DAPMP sample collections, significantly improved procedures for the shipment, storage and primary extraction of soil and sediment samples were initiated. Shipment of soil and sediment samples from the DAPMP installations to US Army Environmental Hygiene Agency/Pest Management and Pesticide Monitoring Division (USAEHA/PMPMD) was expedited by the use of priority US Mail. Upon receipt at USAEHA, sediment samples were immediately logged in and then vacuum filtered in a Buchner funnel for 2-12 hours to remove gravitational water. After filtering, sediment samples were returned to their collection jars and stored in a refrigerator at 4°C until primary extraction. Soil samples, after receipt, were immediately logged in and then placed in a freezer at -10°C until primary extraction. Primary extractions of sediment and soil samples were usually carried out within 7-10 days and 30 days, respectively, following date of collection. The above described procedures greatly increased the integrity of soil and sediment samples and permitted reasonably valid analyses for nonpersistent pesticides such as the organophosphorus insecticides.

c. Quality Control Guidelines. Analytical quality control procedures employed in the DAPMP are based in theory and application on the principles and guidelines presented in the references listed below.

(1) "Manual of Analytical Quality Control for Pesticides and Related Compounds in Human and Environmental Samples," US Environmental Protection Agency (EPA), Health Effects Research Laboratory (HERL), Research Triangle Park (RTP), NC, EPA-600/1-79-008, January 1979.

(2) "Guidelines on Analytical Methodology for Pesticide Residue Monitoring," Federal Working Group on Pest Management, Washington, DC, June 1975.

(3) "Guidelines on Sampling and Statistical Methodologies for Ambient Pesticide Monitoring," Federal Working Group on Pest Management, Washington, DC, October 1974.

2. CRITERIA FOR ANALYTICAL PRECISION AND ACCURACY.

a. Criteria for analytical precision and accuracy for the DAPMP are specified in Appendix F of USAEHA Regulation 702-1, USAEHA Quality Assurance Program, 16 February 1979.

b. Acceptable levels for Percent Relative Standard Deviation (% RDS) in routinely employed DAPMP analytical methodology are shown in Table B-1.

c. Acceptable limits for the Standard Error Unit* parameter in routinely employed DAPMP analytical methodology are:

$$\pm 3 \text{ SEU}$$

(1) If the SEU parameter is between ±2 SEU, the data are acceptable and no action is necessary.

(2) If the SEU parameter is outside ±2 SEU but inside ±3 SEU, the data are considered acceptable; however, the data are reviewed for possible or potential problems.

(3) If the SEU parameter exceeds ±3 SEU for an analysis, applicable analytical operations are ceased until discrepancies are identified and resolved.

---

* Standard Error Unit (SEU) parameter is calculated as follows:

$$\text{SEU} = \frac{\text{AD}}{\text{SEV}} \frac{\text{(Average Deviation)}}{\text{(Standard Error Value)}}$$

AD = Observed Value (corrected for bias, if necessary) minus Formulation Value

SEV = % RSD X Formulation Value

Part II, Pesticide Montrg Sp Study No. 17-44-0230-81, CY 75-78

TABLE B-1. ACCEPTABLE LEVELS FOR PERCENT RELATIVE STANDARD DEVIATION.

Pesticide	Soil & Sediment Analysis	Fish & Bird Analysis
αB-BHC	10	15
βBHC	10	15
aldrin	10	15
chlordane	12	17
trans-chlordane	10	15
cis-chlordane	10	15
oxychlordane	10	15
o,p' - DDD	10	15
p,p' - DDD	10	15
p,p' - DDE	10	15
o,p' - DDE	10	15
o,p' - DDT	10	15
o,p' - DDT	10	15
dieldrin	10	15
endrin	10	15
heptachlor	10	15
heptachlor epoxide	10	15
lindane	10	15
methoxychlor	10	15
mirex	10	15
toxaphene	12	17
hexachlorobenzene	12	17
Aroclors® 1248, 1254 and 1260	12	17
chlorpyrifos	10	15
ronnel	10	15
diazinon	10	15
malathion	10	15
methyl parathion	10	15
parathion	10	15

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3. INTRALABORATORY QUALITY CONTROL PROCEDURES

a. Use of Intralaboratory Spiked Reference Materials. Intralaboratory Spiked Reference Materials (SPRM) were used in the DAPMP to provide a continuous monitor of the performance capability of each analyst or analyst group and to assess the overall day-to-day quality of laboratory performance.

(1) Soil and Sediment SPRM. Intralaboratory SPRM samples to be used with routine DAPMP soil and sediment analyses were prepared in-house by spiking a requisite number of 150-g subsamples of composited soil (in 1-qt, wide-mouth, glass jars) with varying, known concentrations of six relatively stable organochlorine pesticides. The six compounds used most commonly for the SPRM samples were methoxychlor, dieldrin, lindane, heptachlor epoxide, p,p'-DDT and mirex. The concentrations of the individual pesticides were varied from one batch of SPRM samples to another; the overall concentration range for the six pesticides in the SPRM samples was from 0.02 ppm to 0.50 ppm. After the preparation of each new batch of SPRM samples, from 8-10 randomly selected replicate samples from the batch were analyzed initially by experienced analytical personnel to establish the validity of the SPRM and to generate required baseline statistical data for use with laboratory SPRM quality control charts. The remaining SPRM samples from each batch were stored in a freezer at -10°C until extraction and analysis. Approximately one SPRM sample was run for every 15 routine DAPMP soil and sediment samples.

(2) Fish and Bird SPRM. A supply of chicken fat, fortified with varying known concentrations of 6-7 relatively stable organochlorine pesticides and PCBs, was used as a source of intralaboratory SPRM with routine DAPMP fish and bird analyses. The fortified chicken fat was prepared and periodically distributed (i.e., approximately every 6-9 months) by the Quality Assurance Section, ETD/HERL, EPA, RTP, NC. Receipt of EPA fortified fat for use in the DAPMP was coordinated through the Analytical Quality Assurance Office (AQAO)/USAEHA. The concentrations of pesticides and PCBs in the fortified fat typically ranged from about 0.10-0.20 ppm to 2.00-3.00 ppm. After receipt of each new supply of EPA chicken fat SPRM, approximately six replicate subsamples were analyzed initially by experienced analytical personnel to generate required baseline statistical data for use with laboratory SPRM quality control charts. The supply of fortified chicken fat was stored in a freezer at -10°C when not in use. Approximately one EPA fat SPRM sample was run for every 10 routine DAPMP fish and bird samples. NOTE: On occasion, the EPA fortified fat was submitted as a blind sample for dual use as an interlaboratory round robin quality control sample (see paragraph 4a of this Appendix for a discussion of this use) and as a regular supply for intralaboratory SPRM samples.

(3) SPRM Data Management and Quality Control Charts. After completion of analyses of each SPRM sample, the results were documented by the analyst in a special Intralaboratory Quality Control Data log book. In

addition, the analytical results for each SPRM sample were calculated and plotted on SPRM intralaboratory quality control charts. Separate quality control charts were maintained for soil and sediment SPRM samples and for EPA fortified fat SPRM samples. These quality control charts were continuously monitored and reviewed by supervisory and senior DAPMP personnel to detect any deviations from acceptable performance based on criteria described in paragraph 2c of this Appendix.

b. Glassware Decontamination Quality Control.

(1) All glassware used in the processing and analysis of DAPMP samples was rinsed with acetone after use and then soaked for a minimum of 4 hours in Chem-Solv® biodegradable laboratory glassware cleaner prior to washing in a Forma-Fury® Model 8698 or 8658 glassware washer.

(2) After washing and air-drying, randomly selected pieces of each type of glassware represented in each glassware load were rinsed with pesticide-grade petroleum ether and the rinses concentrated approximately 100 to 1 in a Kuderna-Danish apparatus. The concentrated glassware rinses were screened using electron-capture gas chromatography (EC-GC) for residual pesticide, PCB, and other relevant contaminants prior to placing the glassware back into laboratory use.

(3) Solvents, chemical reagents (i.e., sodium sulfate, sodium chloride, florisil, silicic acid), glass wool and filter paper routinely prepared and used in the DAPMP were periodically checked using EC-GC for contaminants and interferences.

c. Analytical Pesticide Reference Standards.

(1) Primary analytical grade pesticide standards of the highest purity and quality available were used to prepare reference standard solutions. Sources used for primary pesticide standards are listed below:

(a) EPA Quality Assurance Section, Analytical Chemistry Branch, ETD/HERL, RTP, NC 27711

(b) EPA, Pesticides Reference Standards Section, Chemistry Branch, Registration Division, Washington, DC 20460. [NOTE: Following a brief relocation to Beltsville, MD, this source in 1979 was permanently transferred and merged with the EPA source listed in paragraph (a) above.]

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® Forma-Fury is a registered tradename of Forma Scientific Division of Mallincknodt, Inc., Marietta, OH.

(c) Poly Science Corporation, 6366 Gross Point Road, Miles, IL 60648.

(d) RFR Corporation, Hope, RI 02831.

(2) Reference standard solutions were prepared and stored in accordance with procedures described in Section 3M of the reference listed in paragraph 1c(1) of this Appendix.

d. Calibration and Maintenance of Instrumentation.

(1) In accordance with USAEHA Regulation 750-20, Maintenance of Supplies and Equipment, 8 August 1979, all balances used in the DAPMP were calibrated on a scheduled basis by DA Calibration Team personnel and manufacturers' service personnel.

(2) The majority of analytical instruments used in the DAPMP were covered by yearly preventive maintenance and emergency repair service contracts. Recordkeeping for in-house maintenance and repair activities, repair parts stockage, etc, were carried out in accordance with the requirements of USAEHA Regulation 750-1, Equipment Maintenance, 4 November 1977.

4. INTERLABORATORY QUALITY CONTROL PROCEDURES.

a. EPA Interlaboratory Blind Sample Round Robin Program.

(1) Since 1975, analytical personnel of the DAPMP have actively participated in the interlaboratory blind sample round robin program conducted by the Quality Assurance Section, ETD/HERL, EPA, RTP, NC. Under this program a uniformly prepared blind pesticide/PCB-spiked sample (representing several substrate types, i.e., water, serum and chicken fat) was mailed by EPA approximately once a year to all laboratories participating in the program. Receipt of the samples by DAPMP personnel and subsequent reporting of analytical results back to EPA was coordinated through AQAO.

(2) From 1975 through 1979, five EPA interlaboratory blind samples (two water, two fat, and one serum) were received and analyzed by DAPMP personnel. Performance results by DAPMP personnel on the five samples are summarized in Table B-2:

TABLE B-2. PERFORMANCE RESULTS BY DAPMP PERSONNEL ON FIVE EPA INTERLABORATORY BLIND SAMPLES

Year Sample Analyzed	Substrate	DAPMP Laboratory Performance Score (200 Maximum Possible Score)	Ranking of DAPMP Laboratory/ Total No. Participating Laboratories
1975	Water	195.88	4/46
1976	Water	196.11	13/52
1978	Fat	196.29	2/16
1979	Serum	195.50	7/17
1979	Fat	190.37	4/18

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b. AQAO Quarterly Audit Package Samples.

(1) As part of a continuous external evaluation and review of routine DAPMP soil and sediment analytical procedures, several blind pesticide/PCB-spiked soil samples were prepared each quarter by AQAO and submitted for analysis by DAPMP personnel. Results of these analyses were evaluated by AQAO and reported in writing back to the DAPMP Program Manager.

(2) As part of a continuous external evaluation and review of routine DAPMP fish and bird analytical methodology, an EPA fortified chicken fat sample was analyzed each quarter by DAPMP personnel. The EPA fortified fat was analyzed for the AQAO quarterly audit as both a blind sample (i.e., following initial receipt by AQAO of each new supply of EPA fortified fat) and as a known routinely used intralaboratory SPRM sample. Results of the quarterly audit sample analyses were evaluated by AQAO and reported in writing back to the DAPMP Program Manager.

APPENDIX C

SELECTED CHEMICAL CLASSES AND SUBGROUPS USED IN DATA EVALUATION

Class/SubGroup	Pesticides Included
chlordane group	technical chlordane, *cis*-chlordane, *trans*-chlordane, heptachlor epoxide heptachlor, oxychlordane
DDT group	o,p'-DDT, p-p'-DDT
DDD group	o,p'-DDD, p-p'-DDD
DDE group	o,p'-DDE, p-p'-DDE
Total DDT group	DDT group, DDD group, and DDE group
cyclodiene group	chlordane group, aldrin, dieldrin, endrin
organophosphate group	malathion, chlorpyrifos, diazinon parathion, ronnel
BHC group	α-BHC, β-BHC, lindane

APPENDIX D

FIGURES

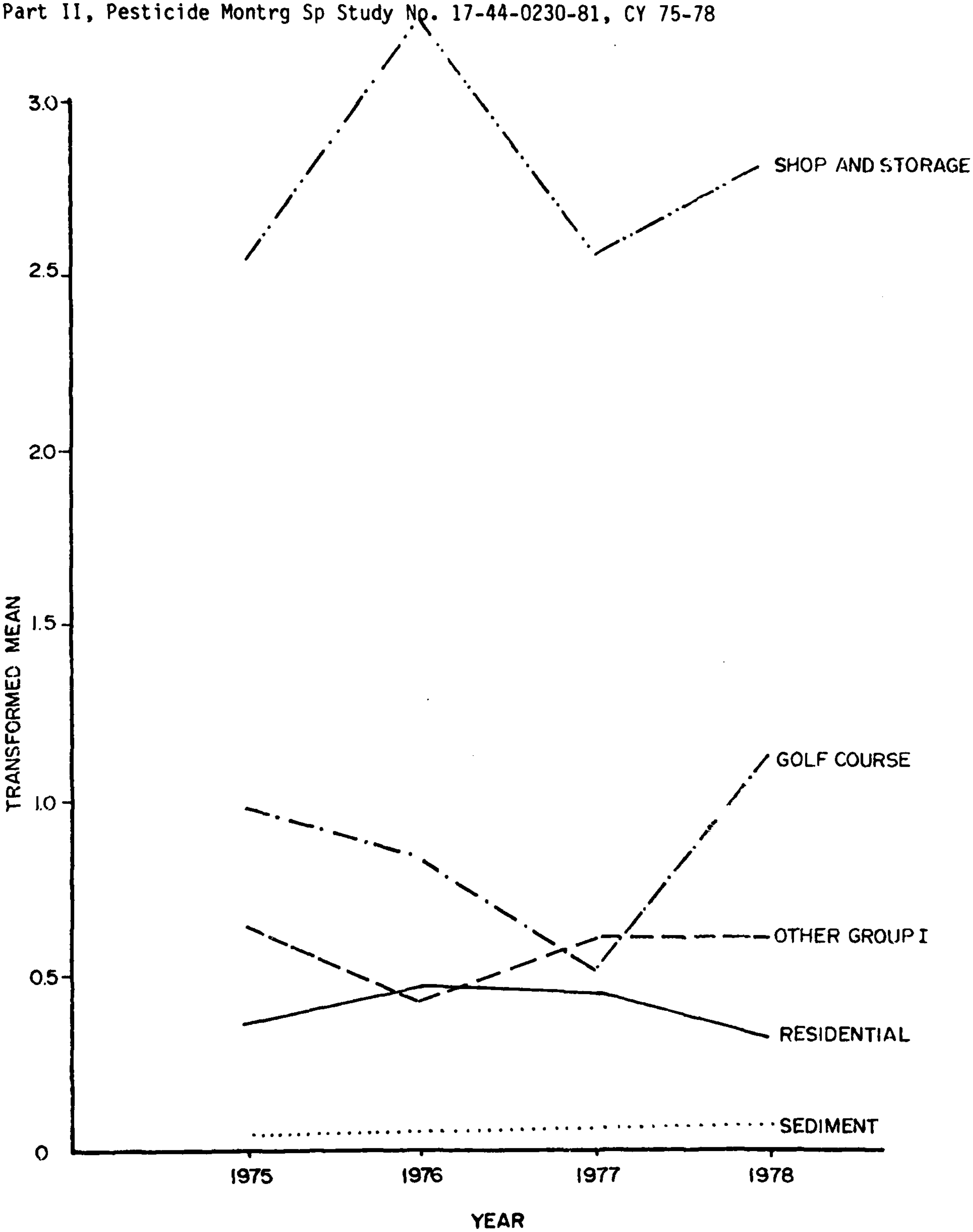


Figure 1. Comparison of Mean Transformed Total By Land Use Areas.

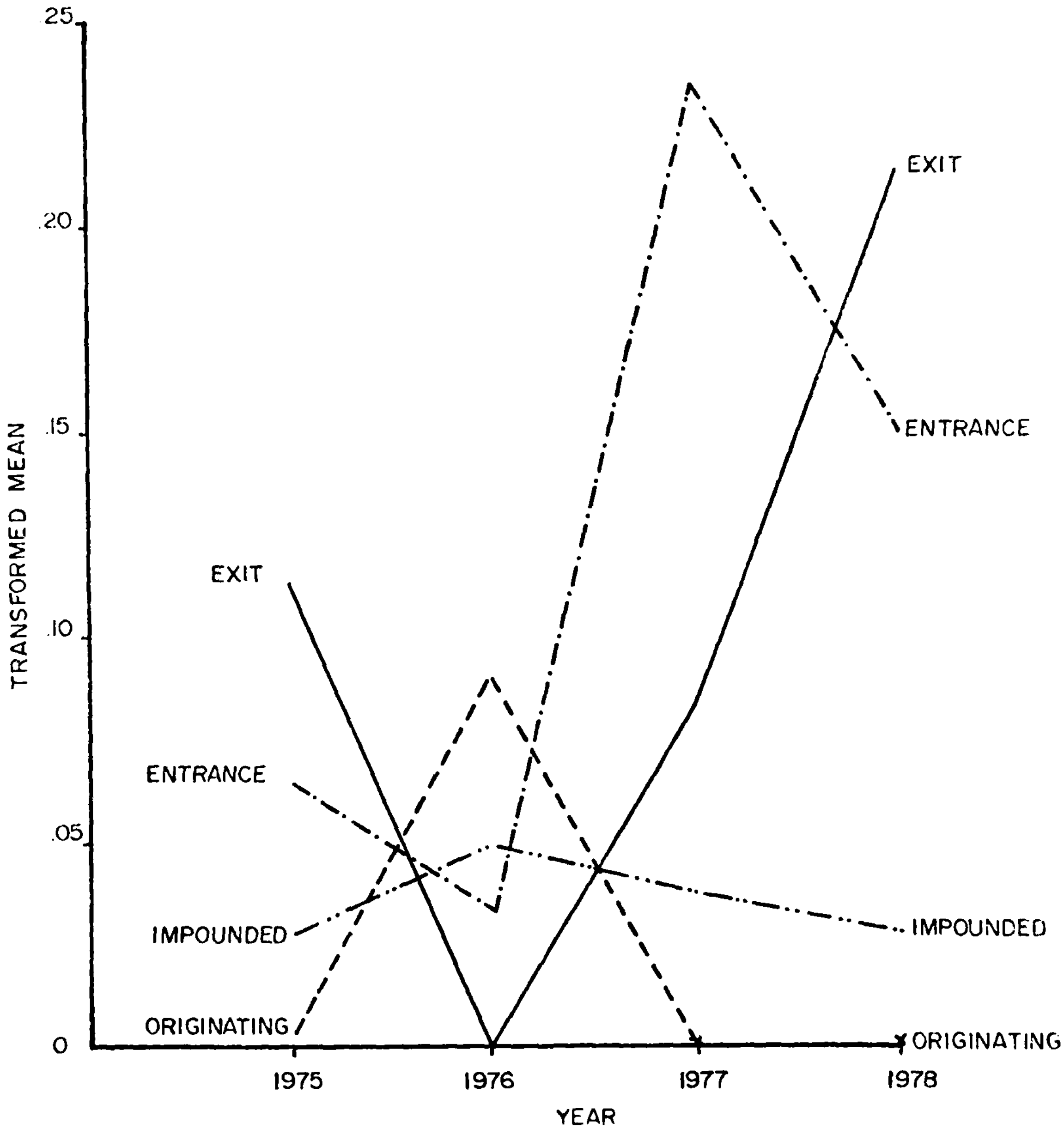


Figure 2. Comparison of Mean Transformed Total in Sediment Sites.

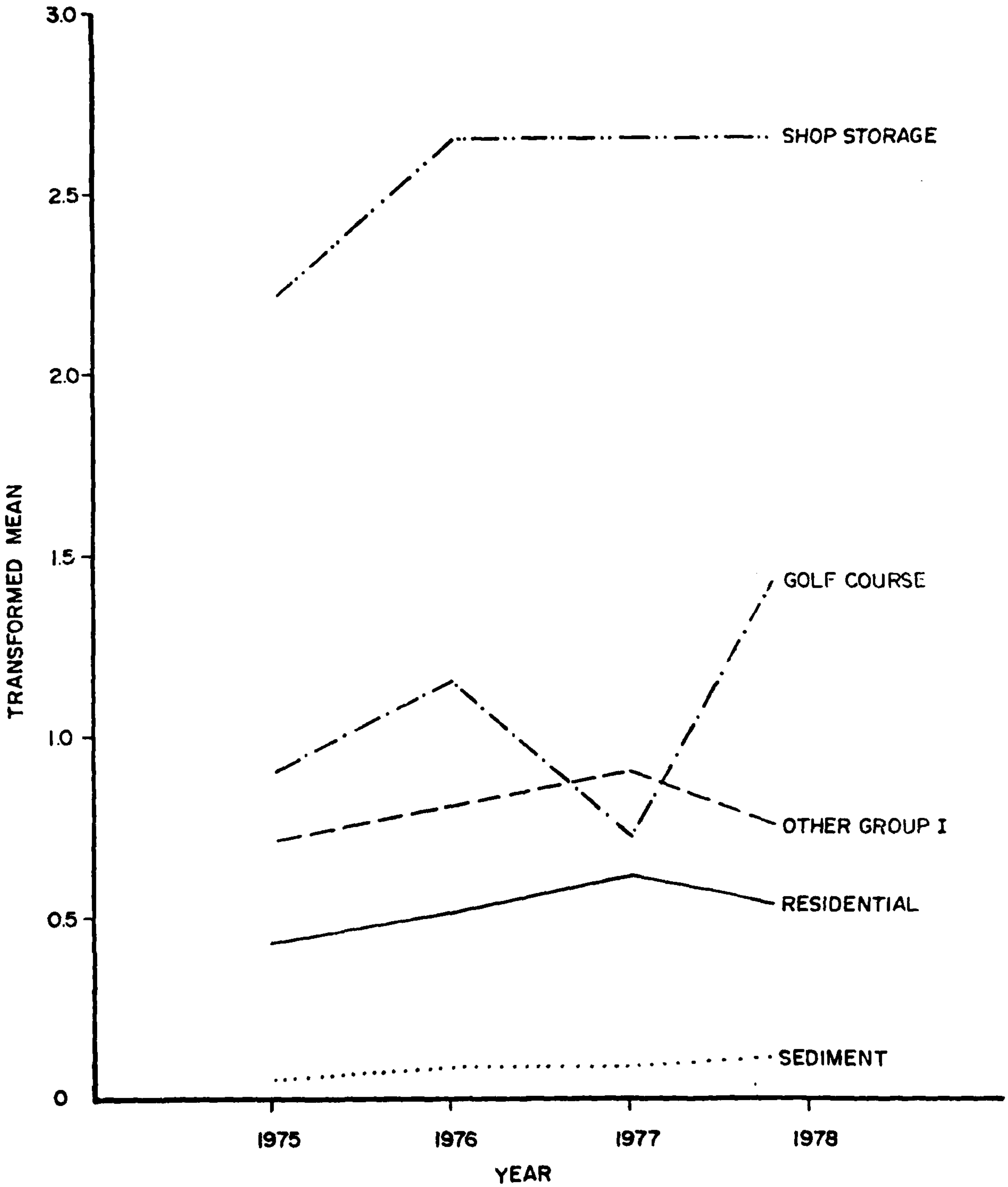


Figure 3. Comparison of the Mean Number of Compounds By Land Use Areas.

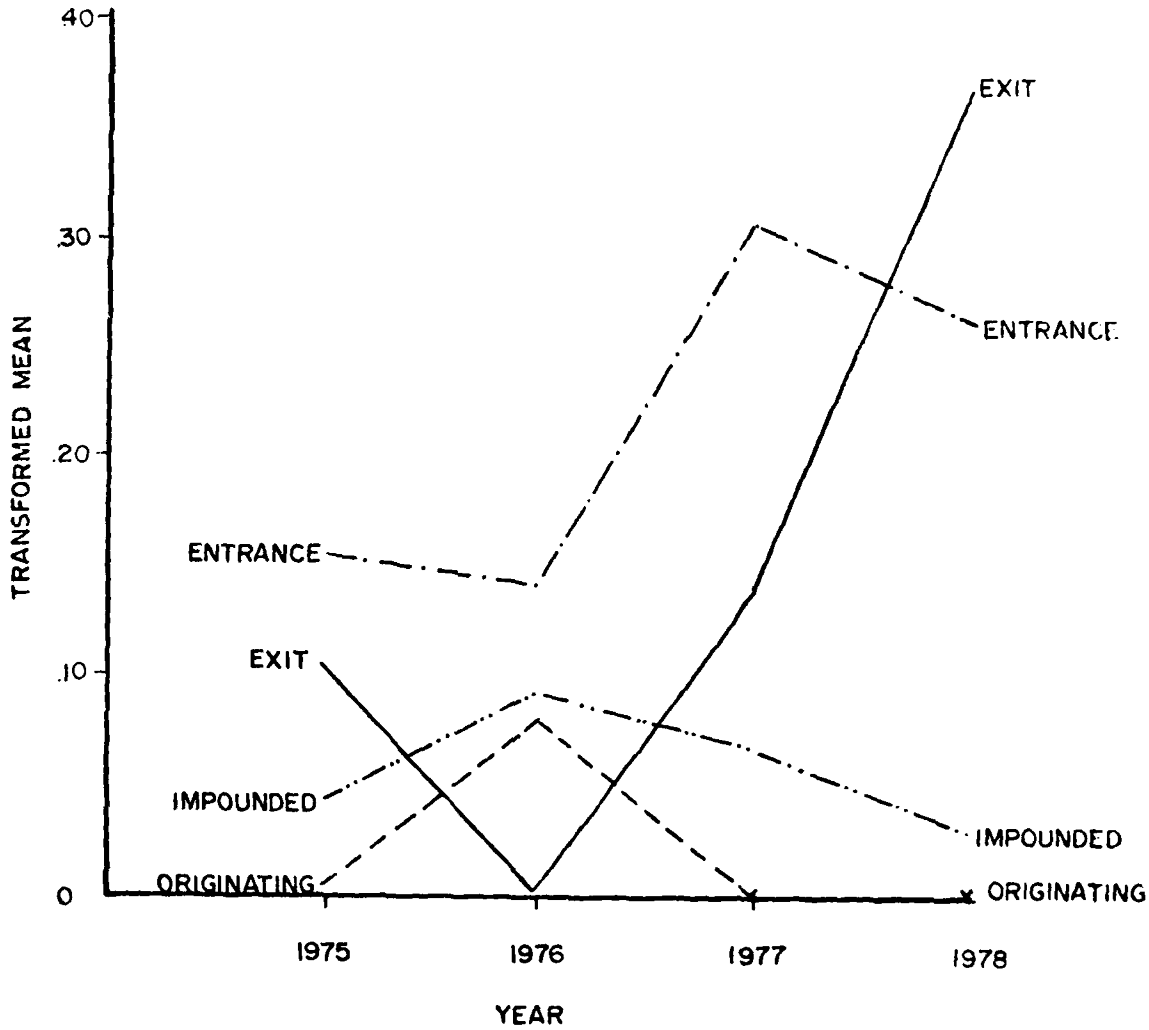


Figure 4. Comparison of the Mean Number of Compounds in Sediment Sites.

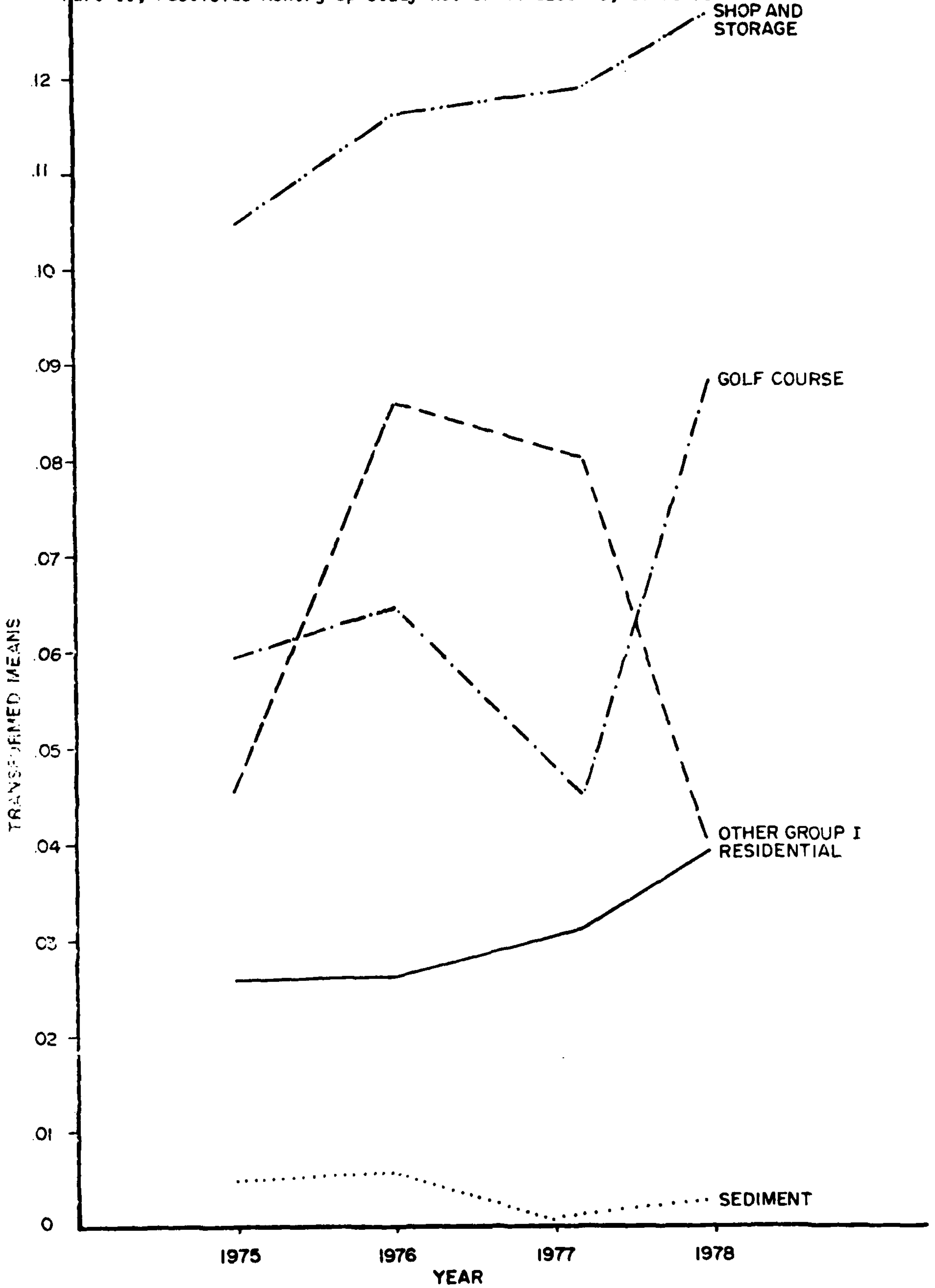
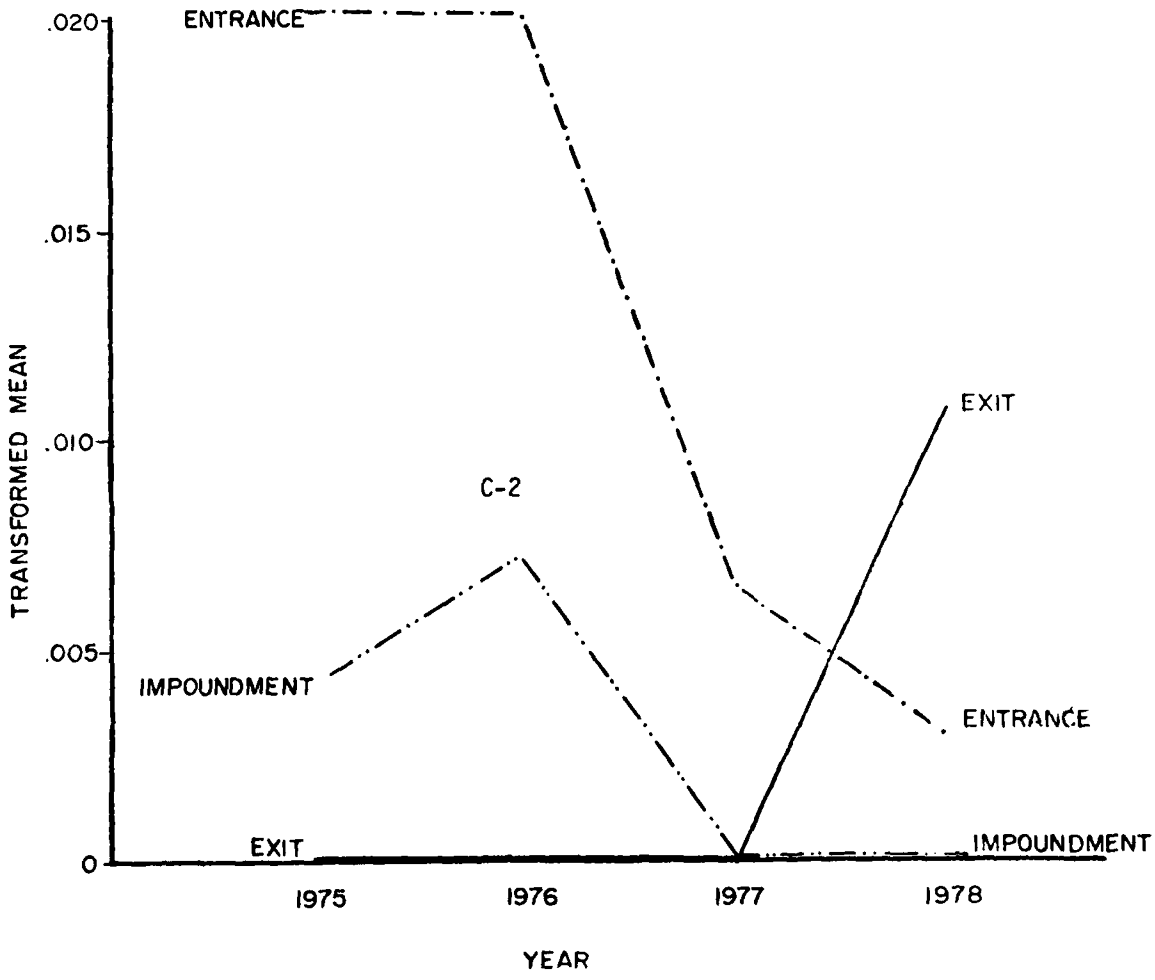


Figure 5. Comparison of Diversity Indices By Land Use Areas.



ORIGINATING = Ø FOR ALL YEARS

Figure 6. Comparison of Diversity Indices in Sediment Sites.

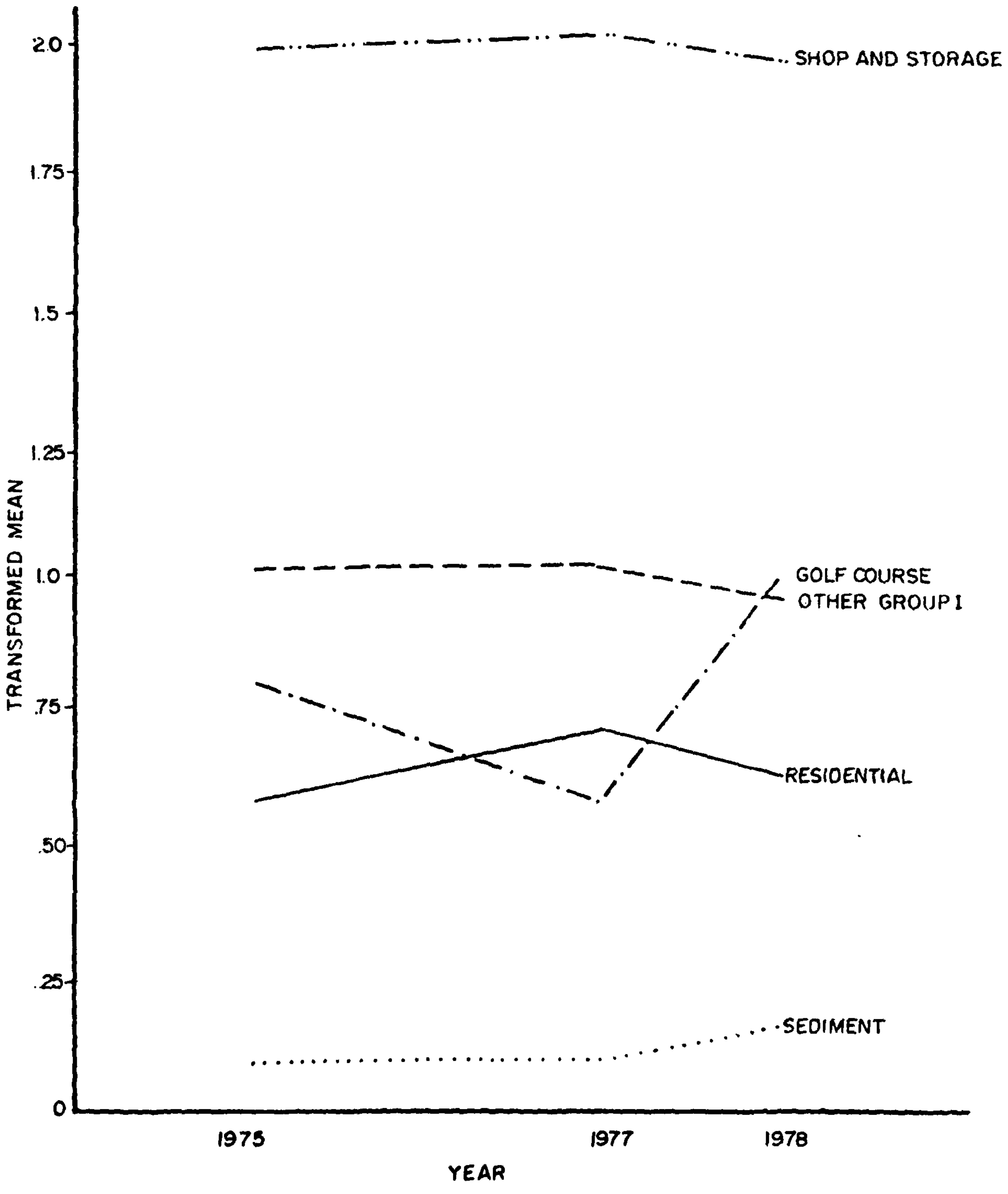


Figure 7. Comparison of Total DDT* Residues By Land Use Stratification.

*Total DDT = DDT + DDE + DDD

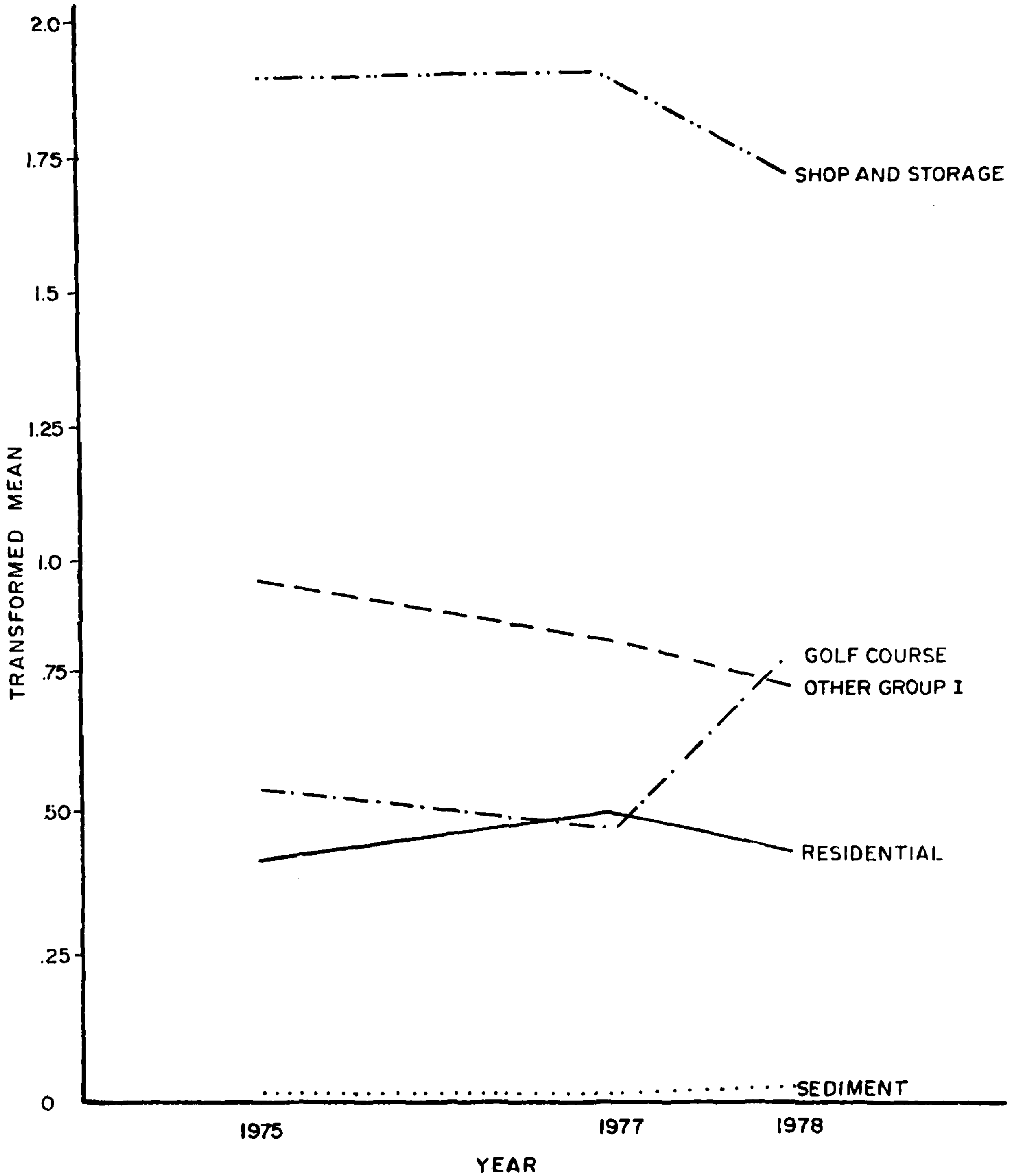


Figure 8. Comparison of Mean DDT Residues By Land Use Stratification.

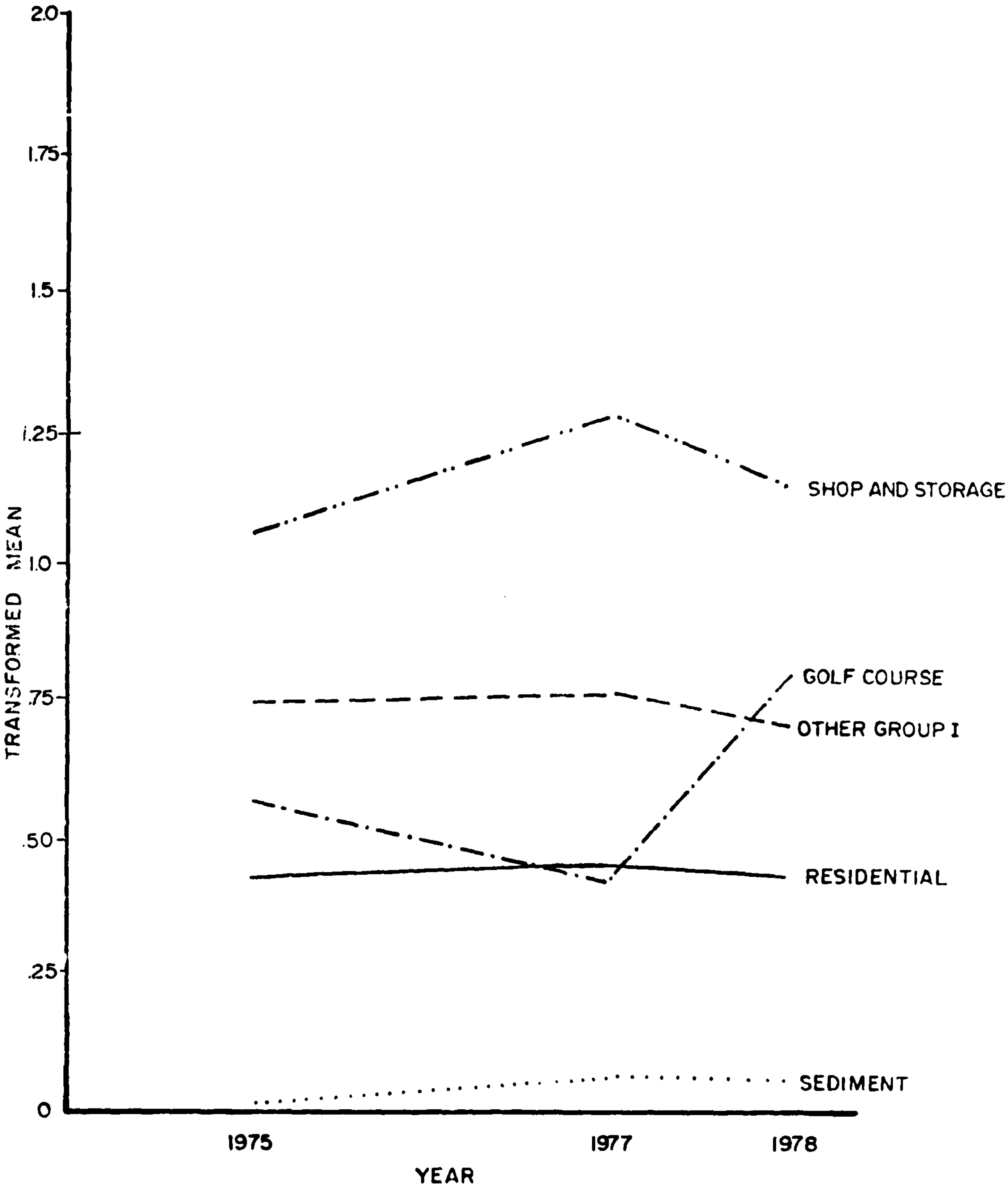


Figure 9. Comparison of Mean DDE Residues By Land Use Stratification.

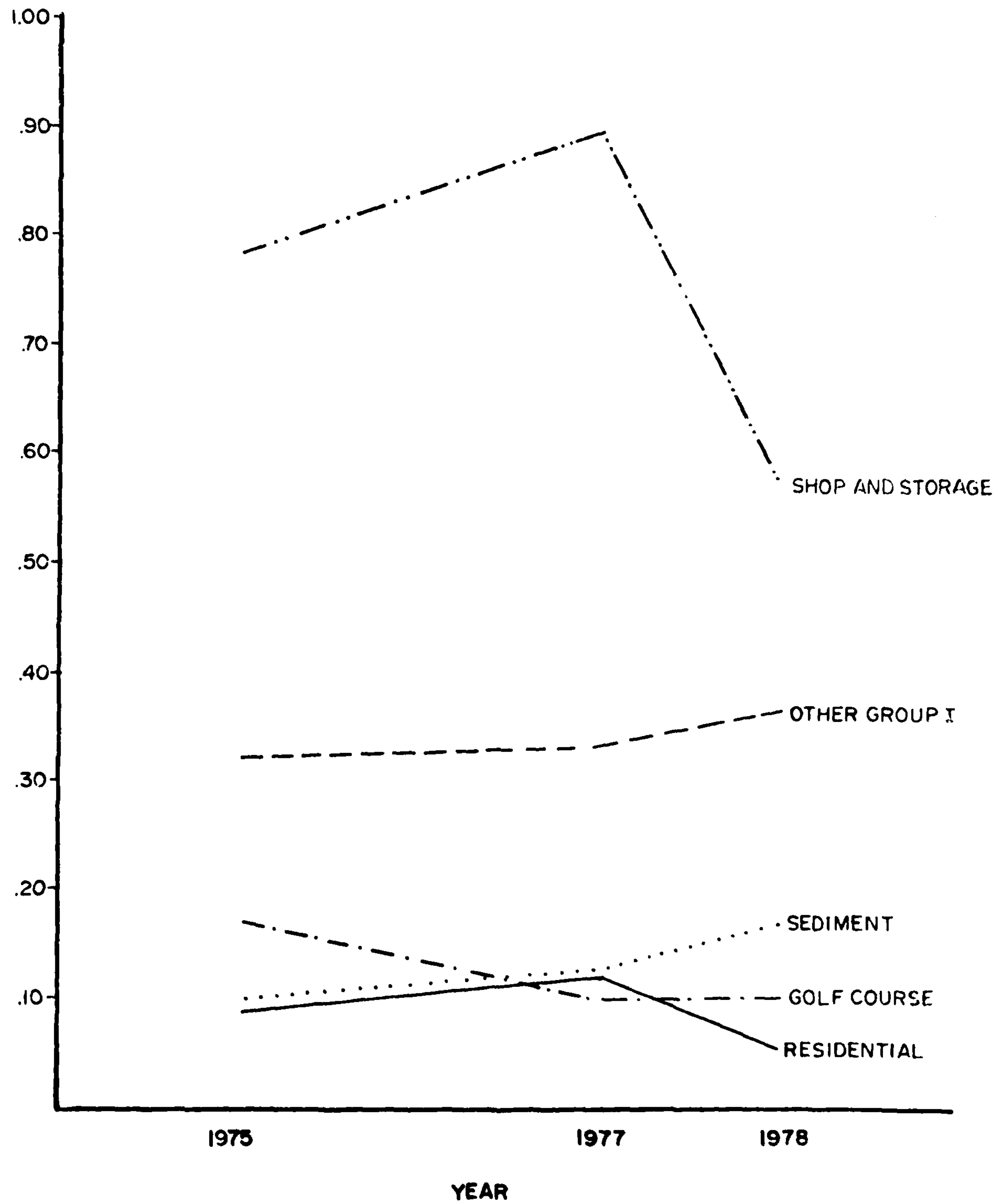


Figure 10. Comparison of Mean DDD Residues By Land Use Stratification.

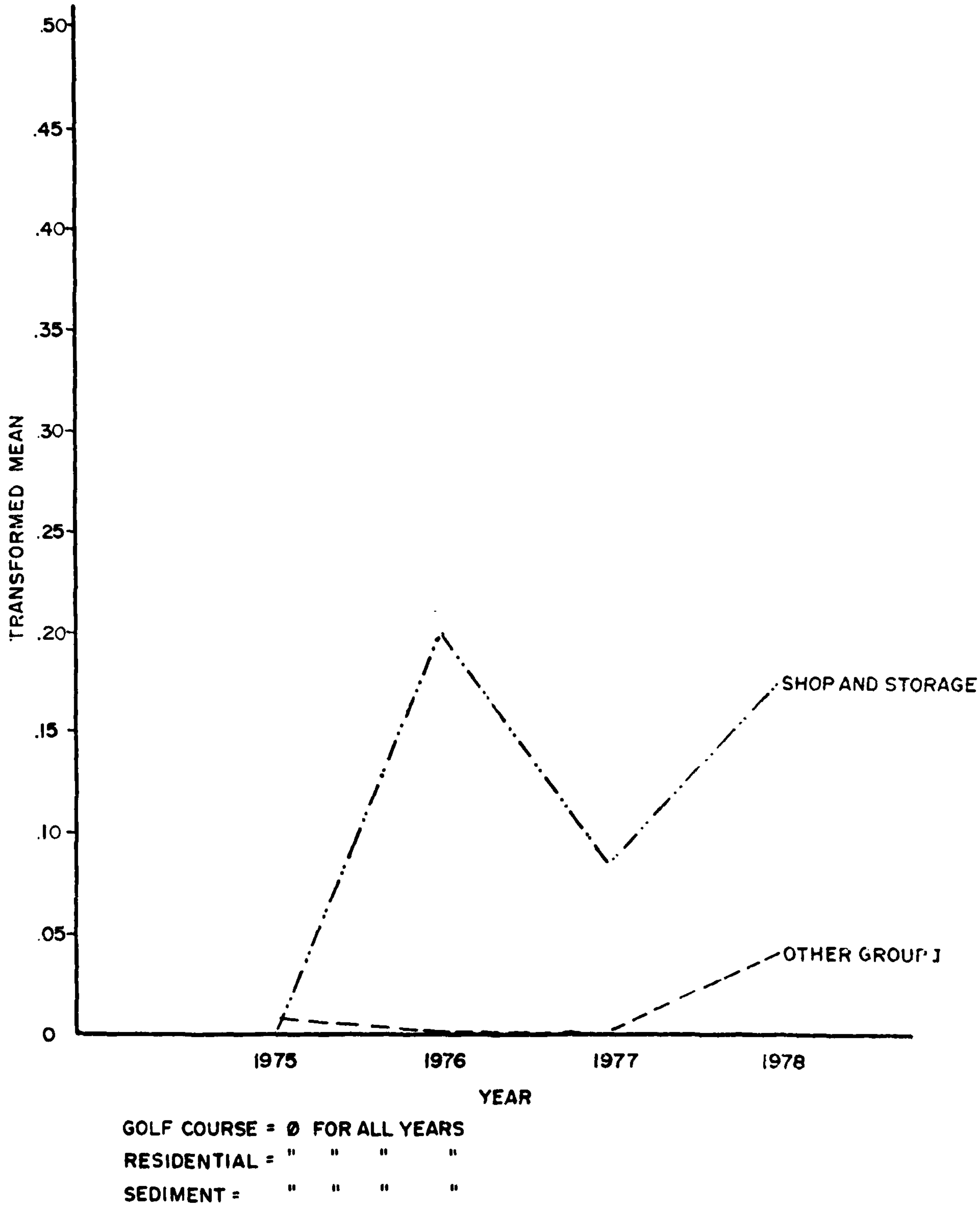


Figure 11. Comparison of Mean BHC Residues By Land Use Stratification.

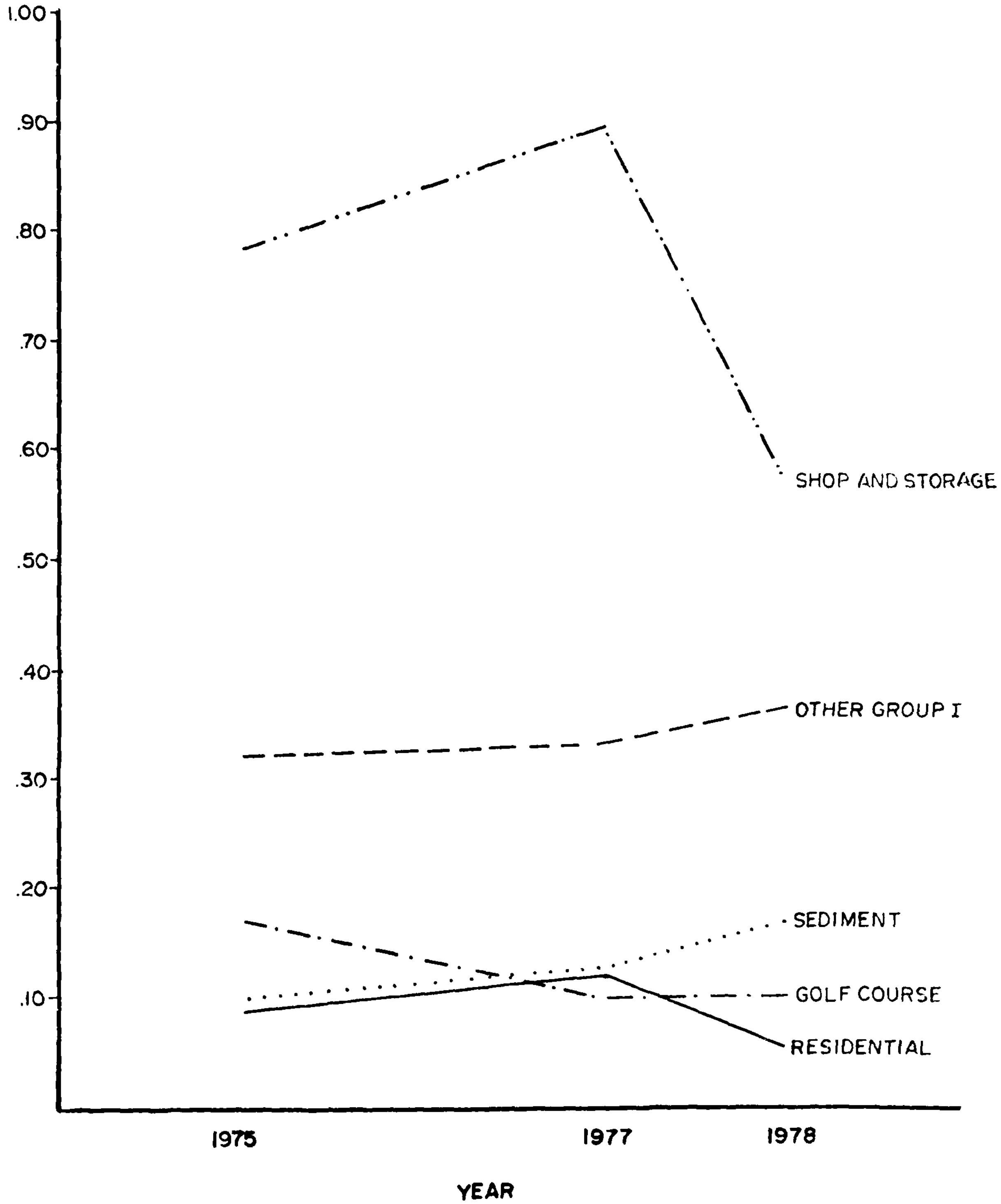
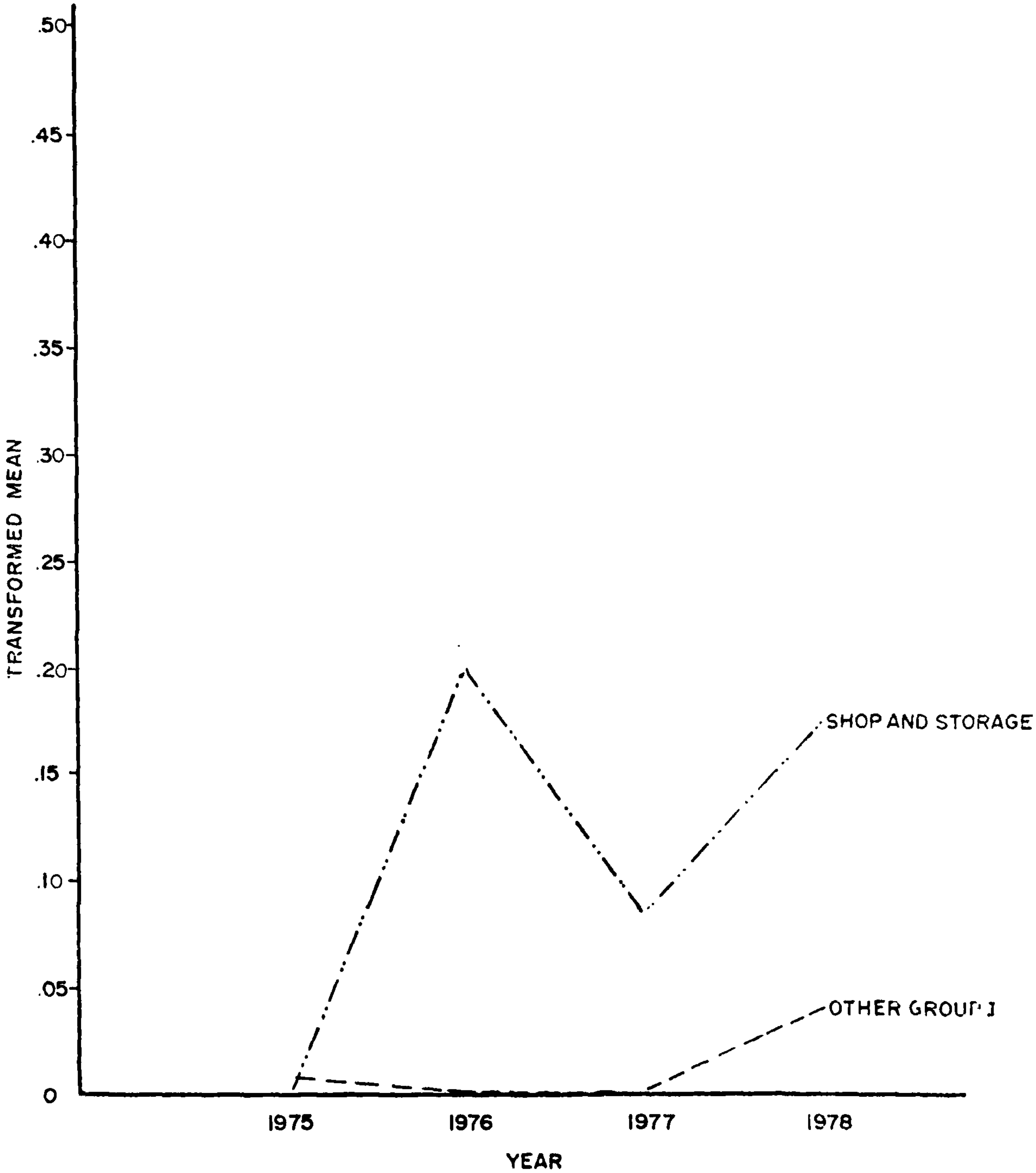


Figure 10. Comparison of Mean DDD Residues By Land Use Stratification.



GOLF COURSE = Ø FOR ALL YEARS
RESIDENTIAL = " " " "
SEDIMENT = " " " "

Figure 11. Comparison of Mean BHC Residues By Land Use Stratification.

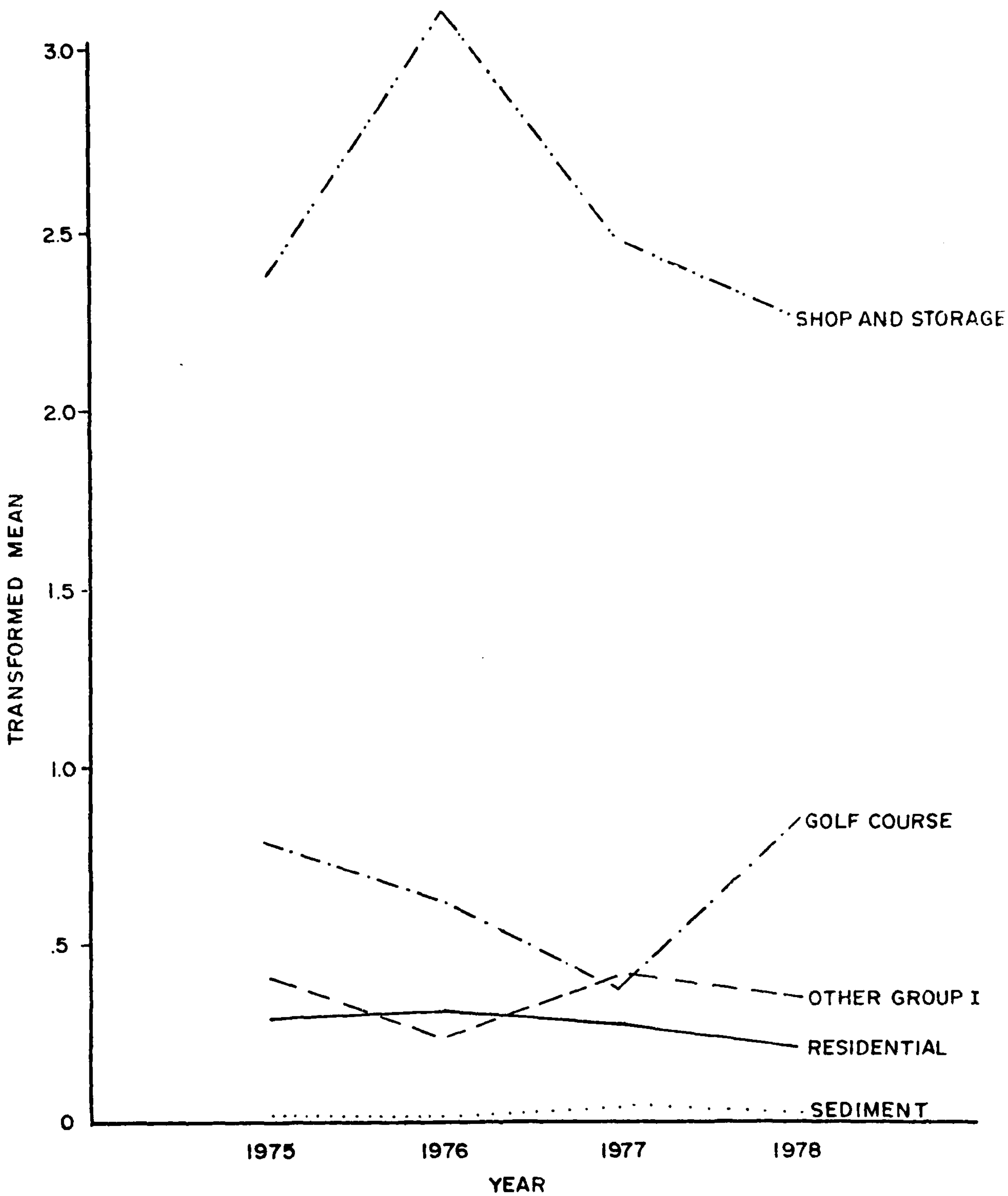


Figure 12. Comparison of Mean Chlordane Residues By Land Use Stratification.

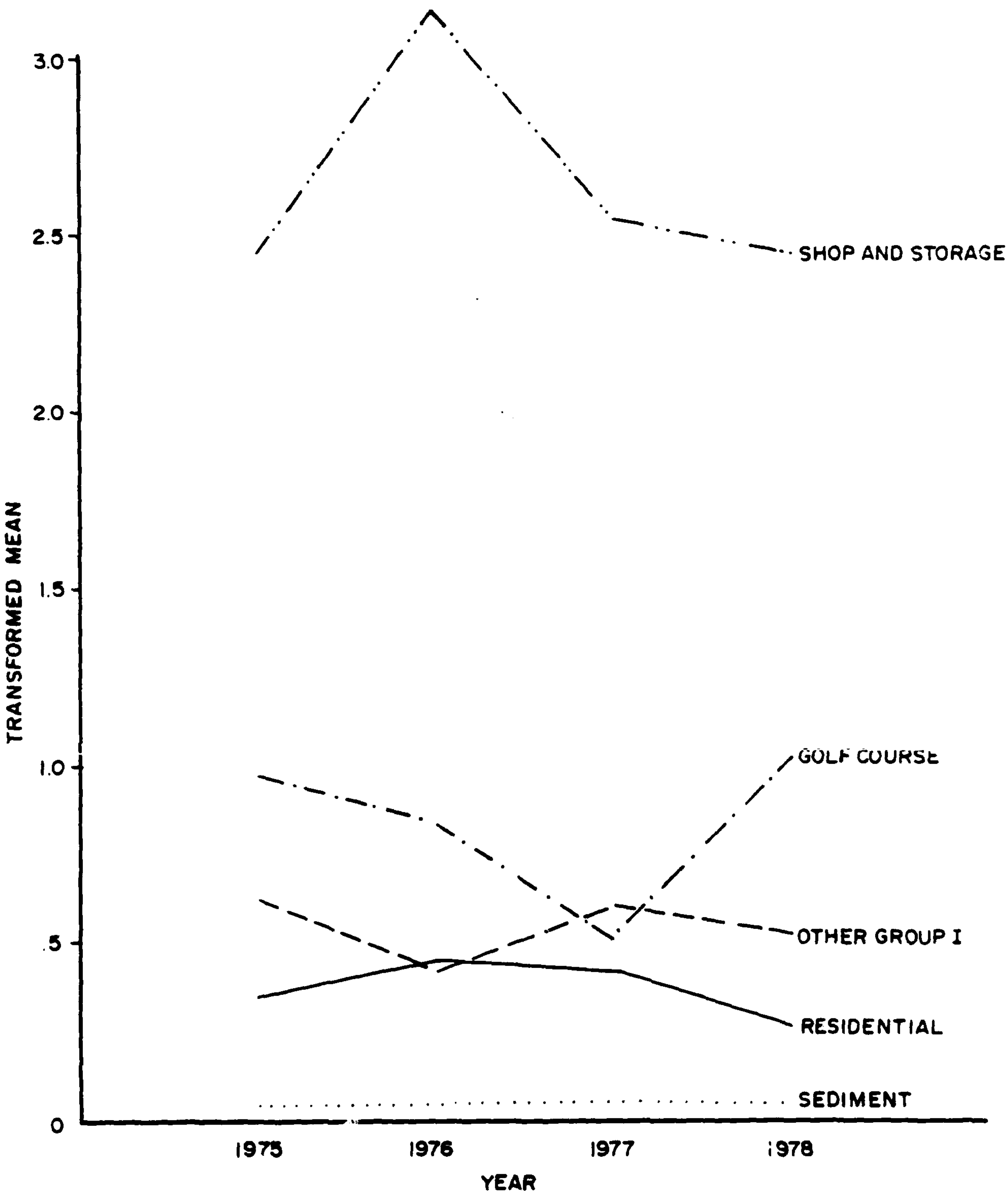


Figure 13. Comparison of Mean Cyclodiene Residues by Land Use Stratification.

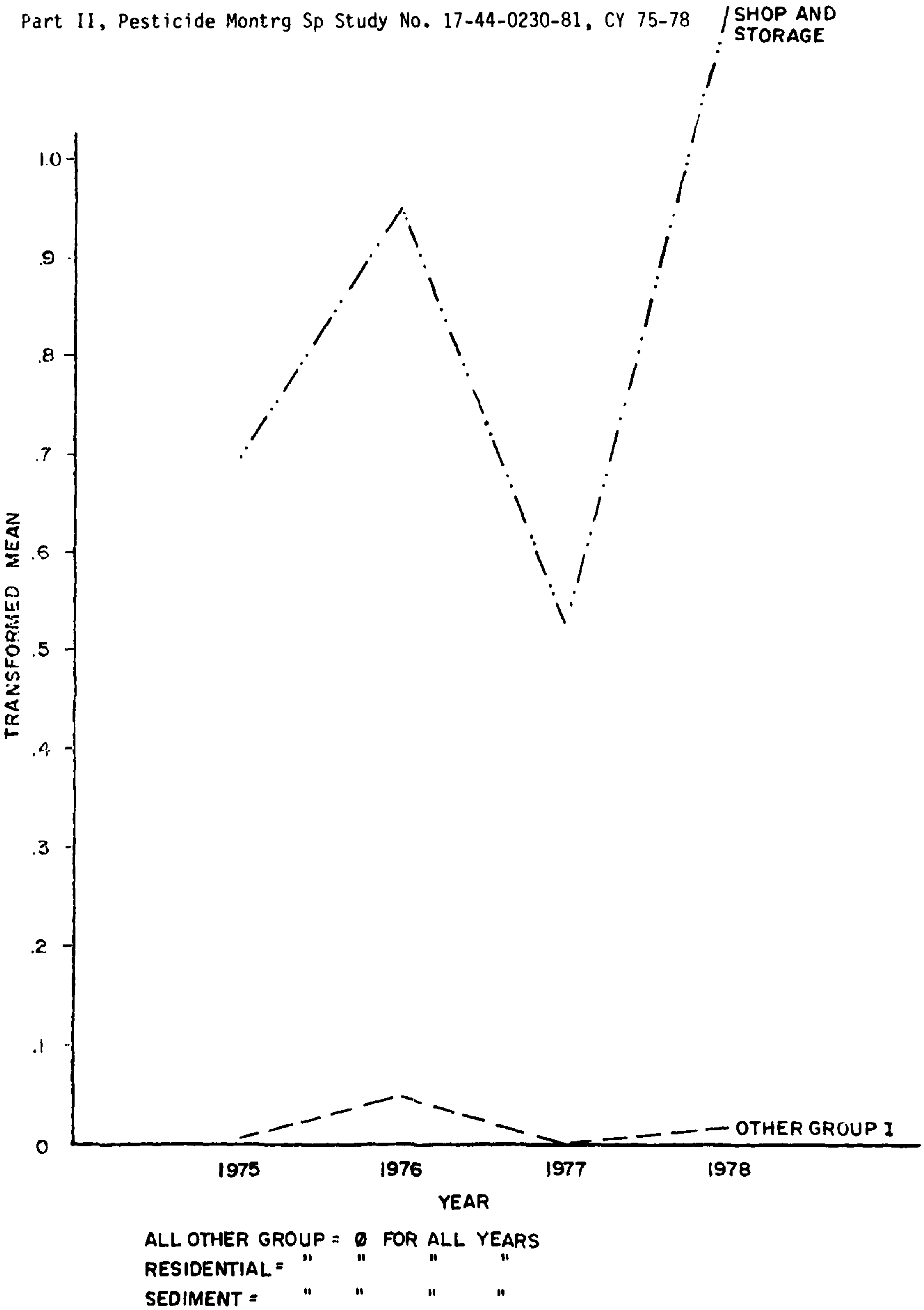


Figure 14. Comparison of Mean Organophosphate Residues By Land Use Stratification.

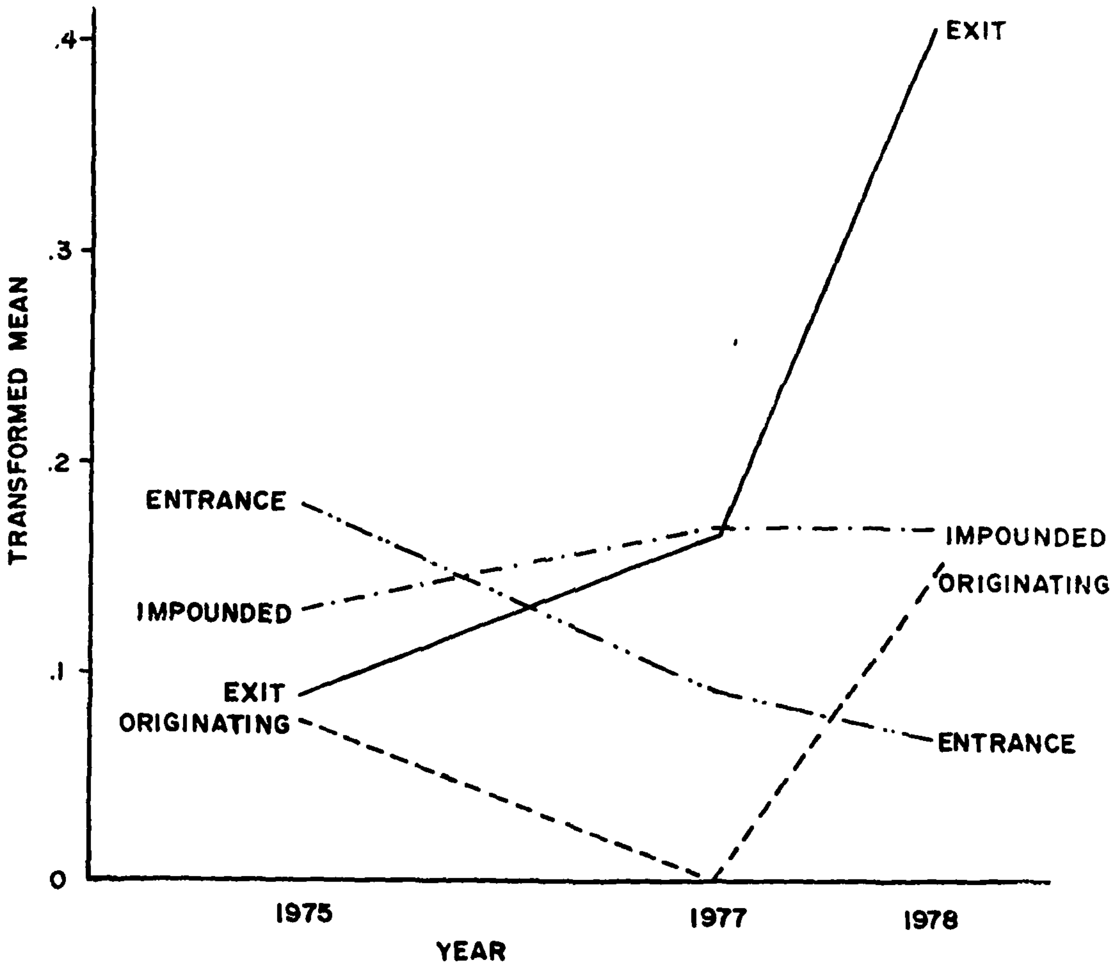


Figure 15. Comparison of Mean Total DDT* Residues by Sediment Stratification.

* Total DDT = DDT + DDE + DDD

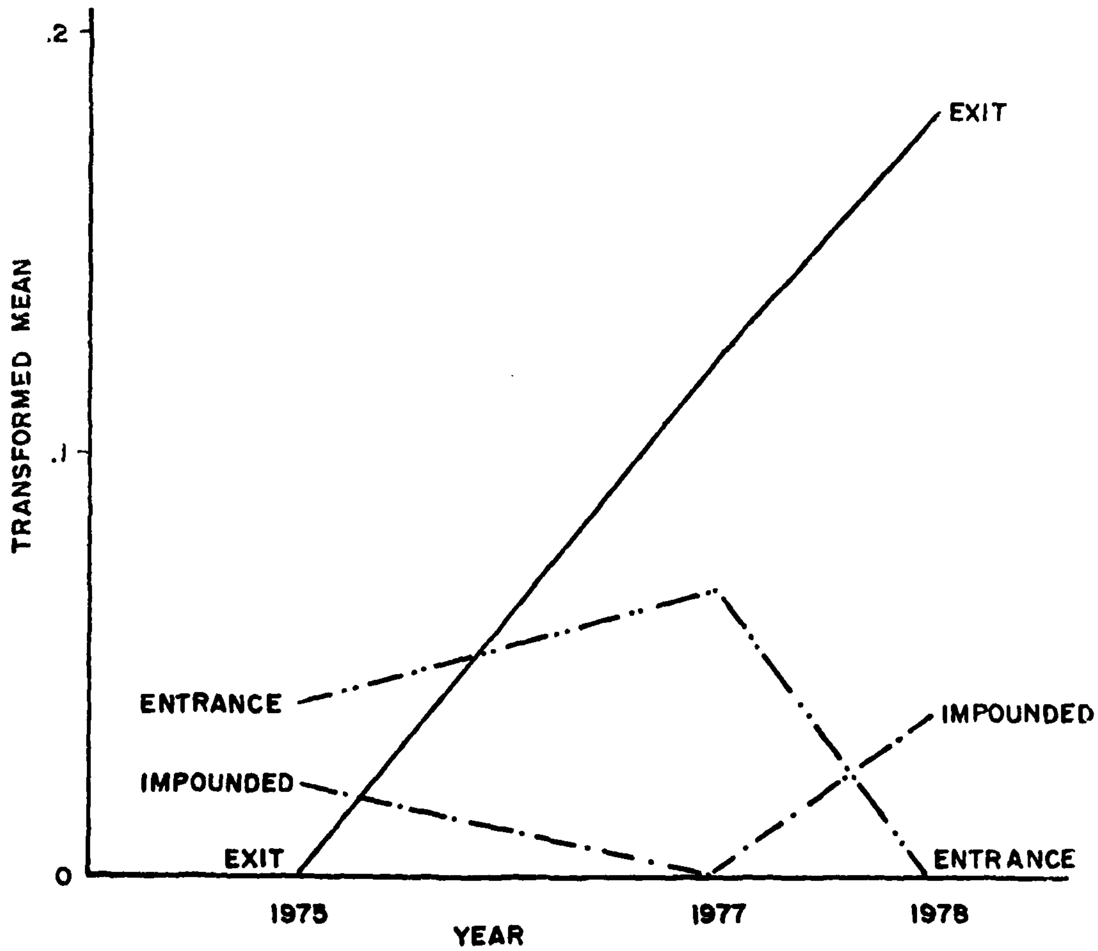


Figure 16. Comparison of Mean DDT Residues by Sediment Stratification.

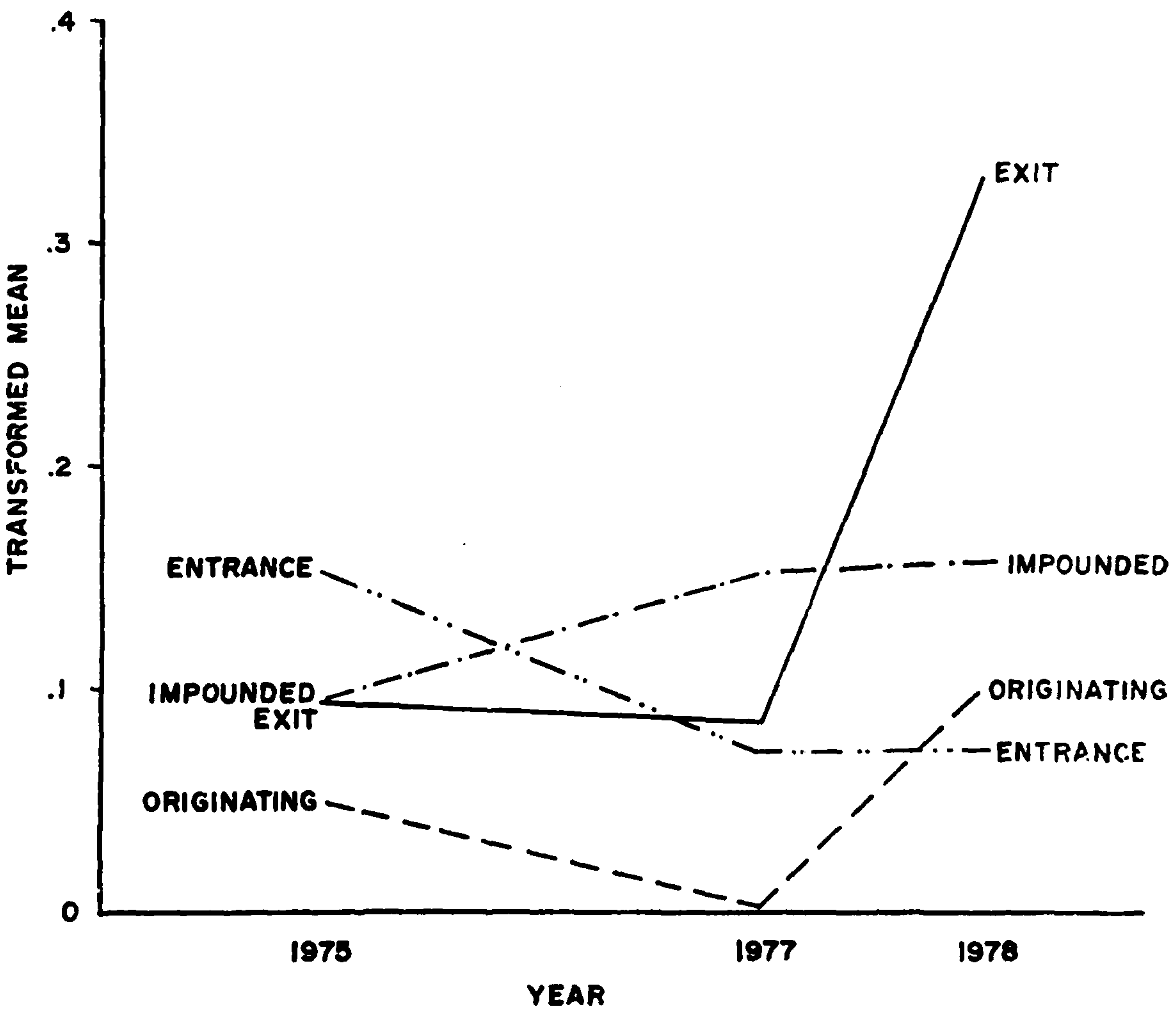


Figure 17. Comparison of Mean DDD Residues By Sediment Stratification.

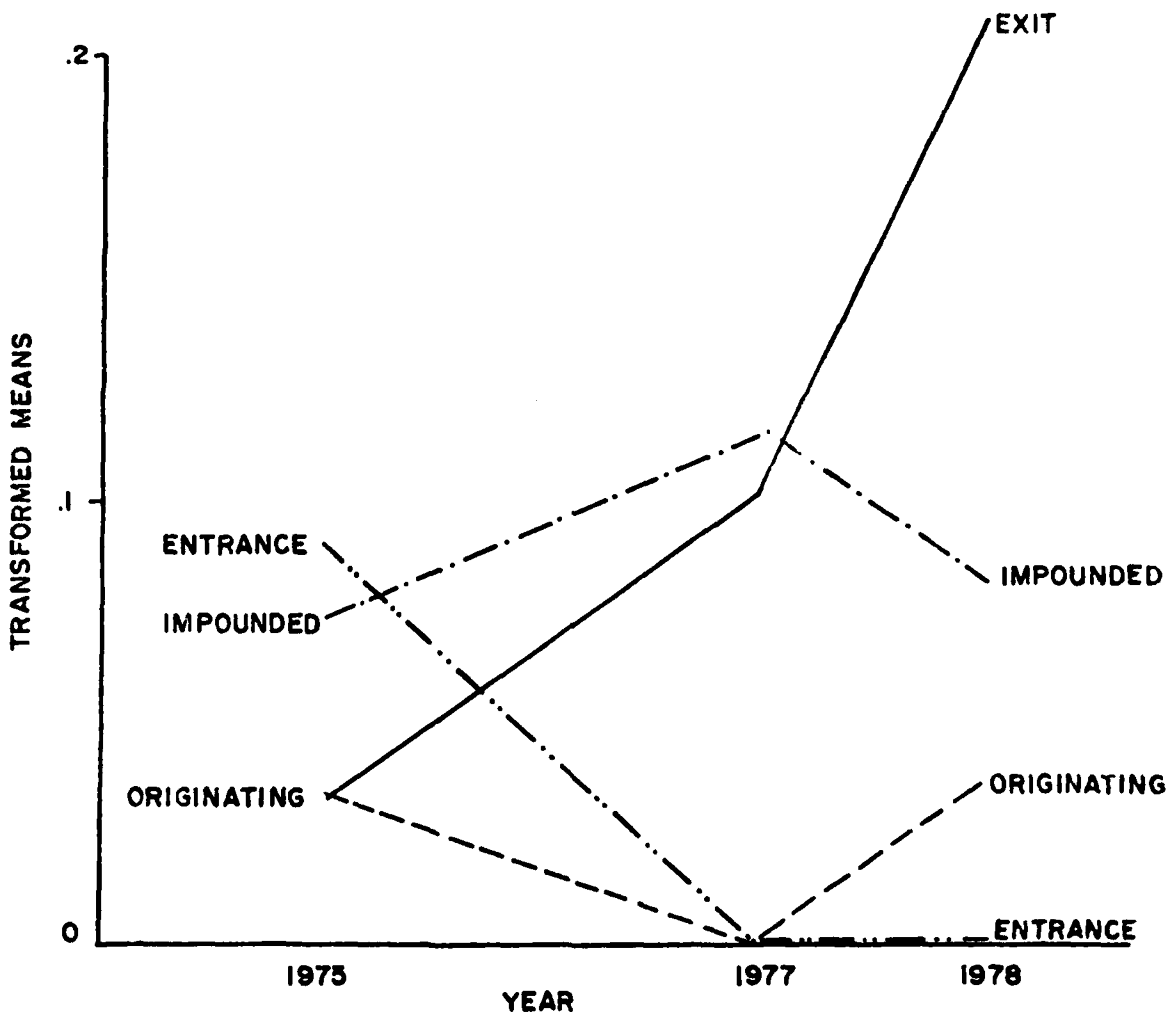


Figure 18. Comparison of Mean DDE Residues By Sediment Stratification.

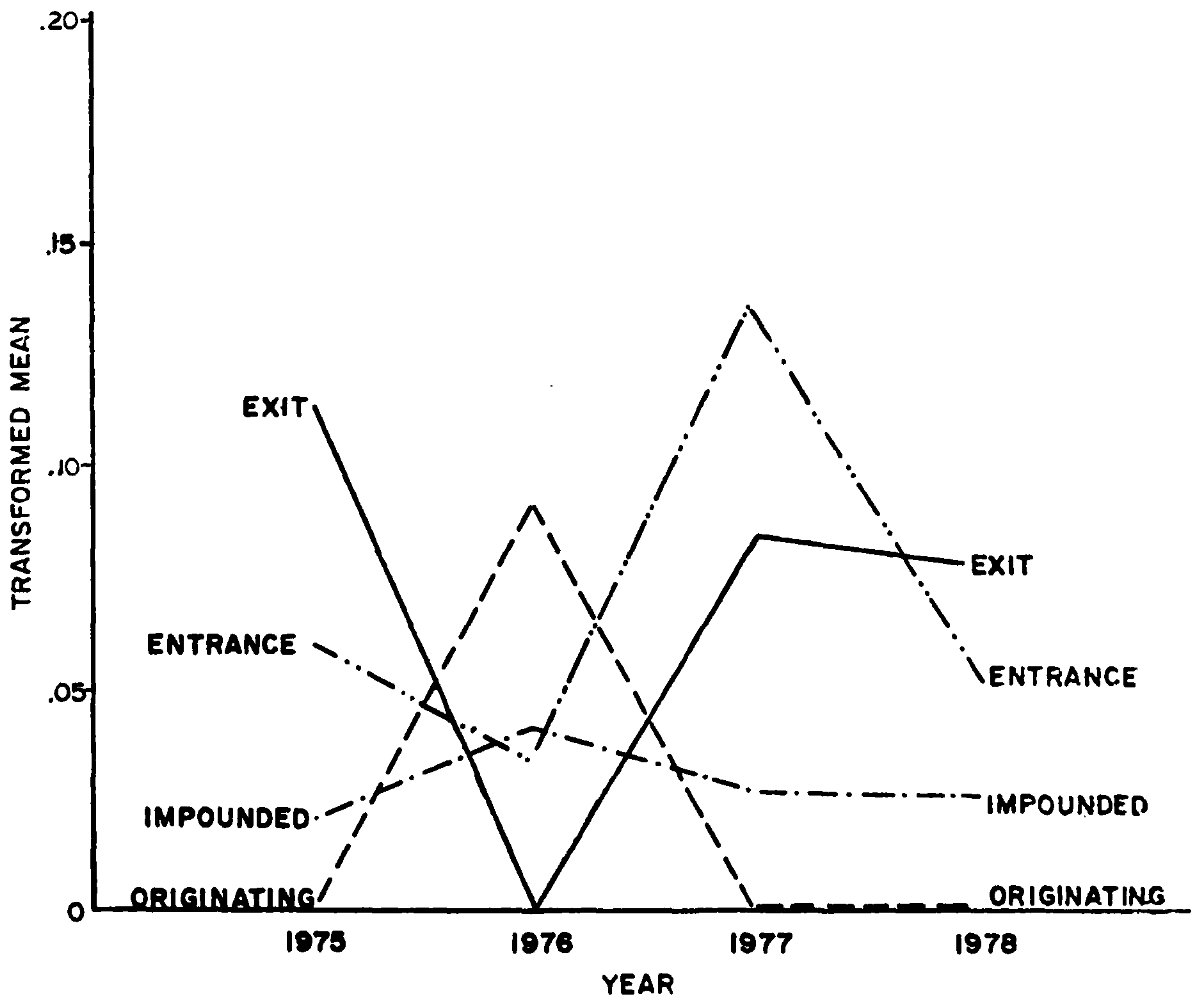


Figure 19. Comparison of Mean Chlordane Residues By Sediment Stratification.

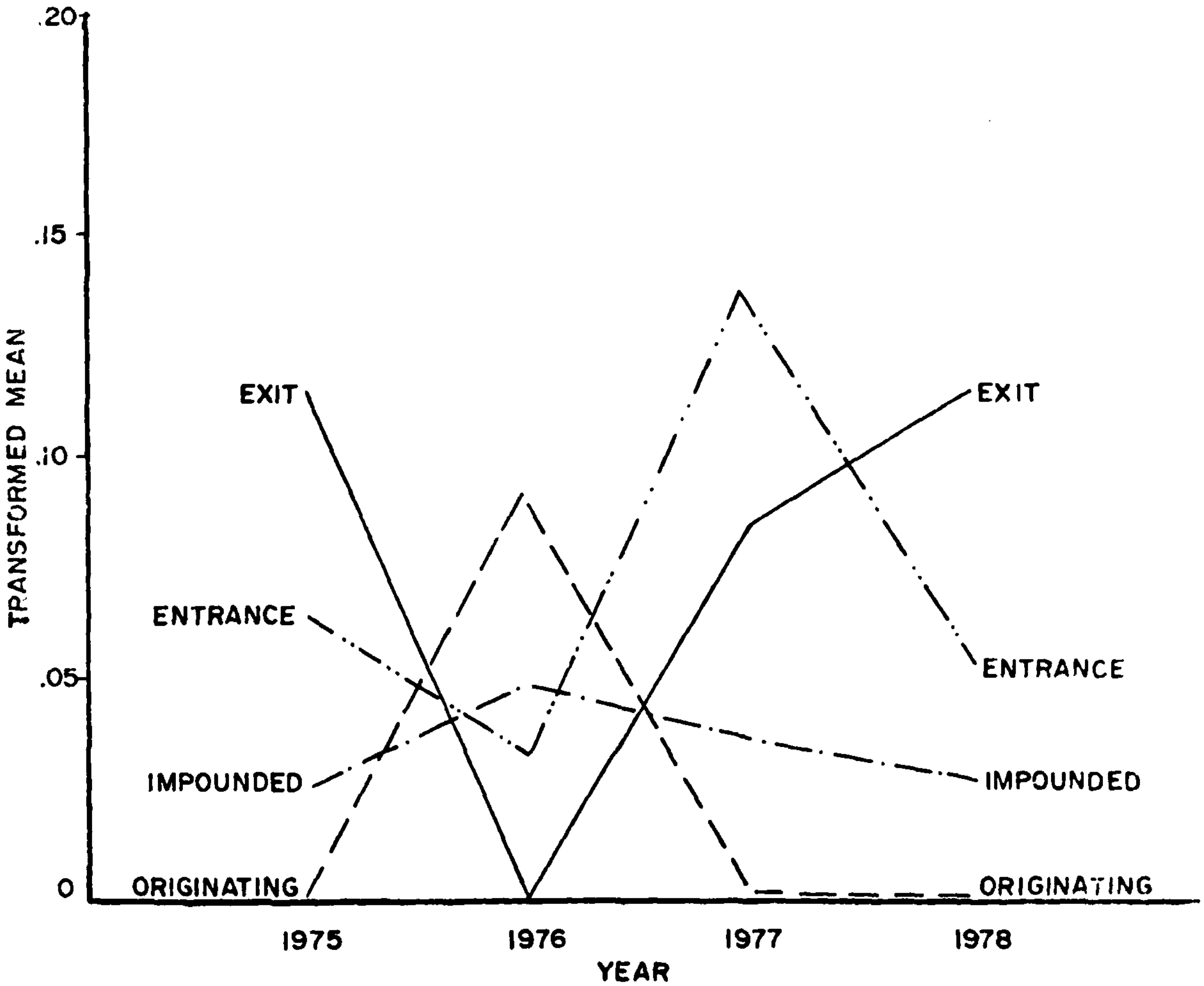


Figure 20. Comparison of Mean Cyclodiene Residues By Sediment Stratification.

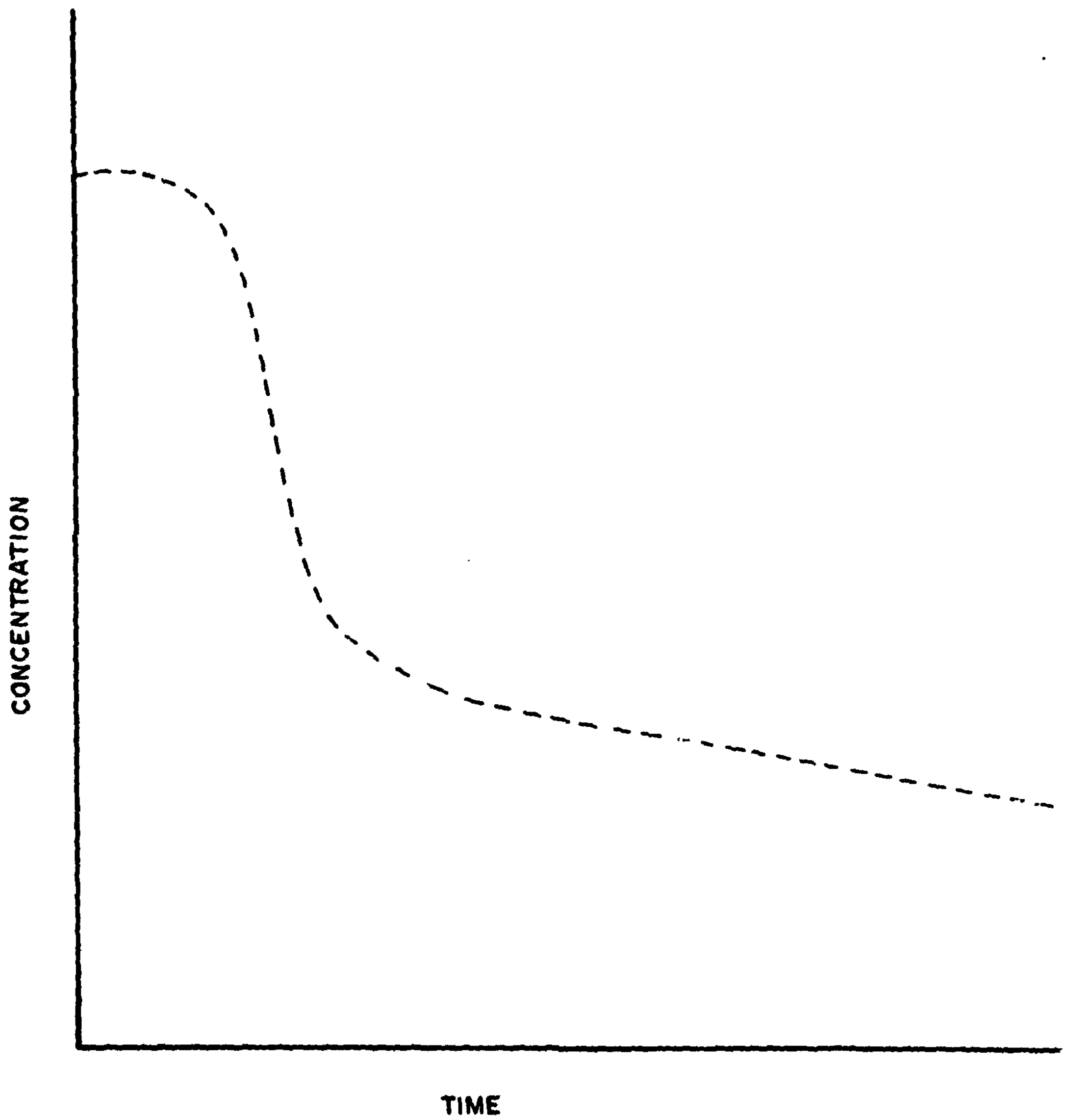


Figure 21. A Typical Hypothetical Persistent Pesticide Degradation Curve.

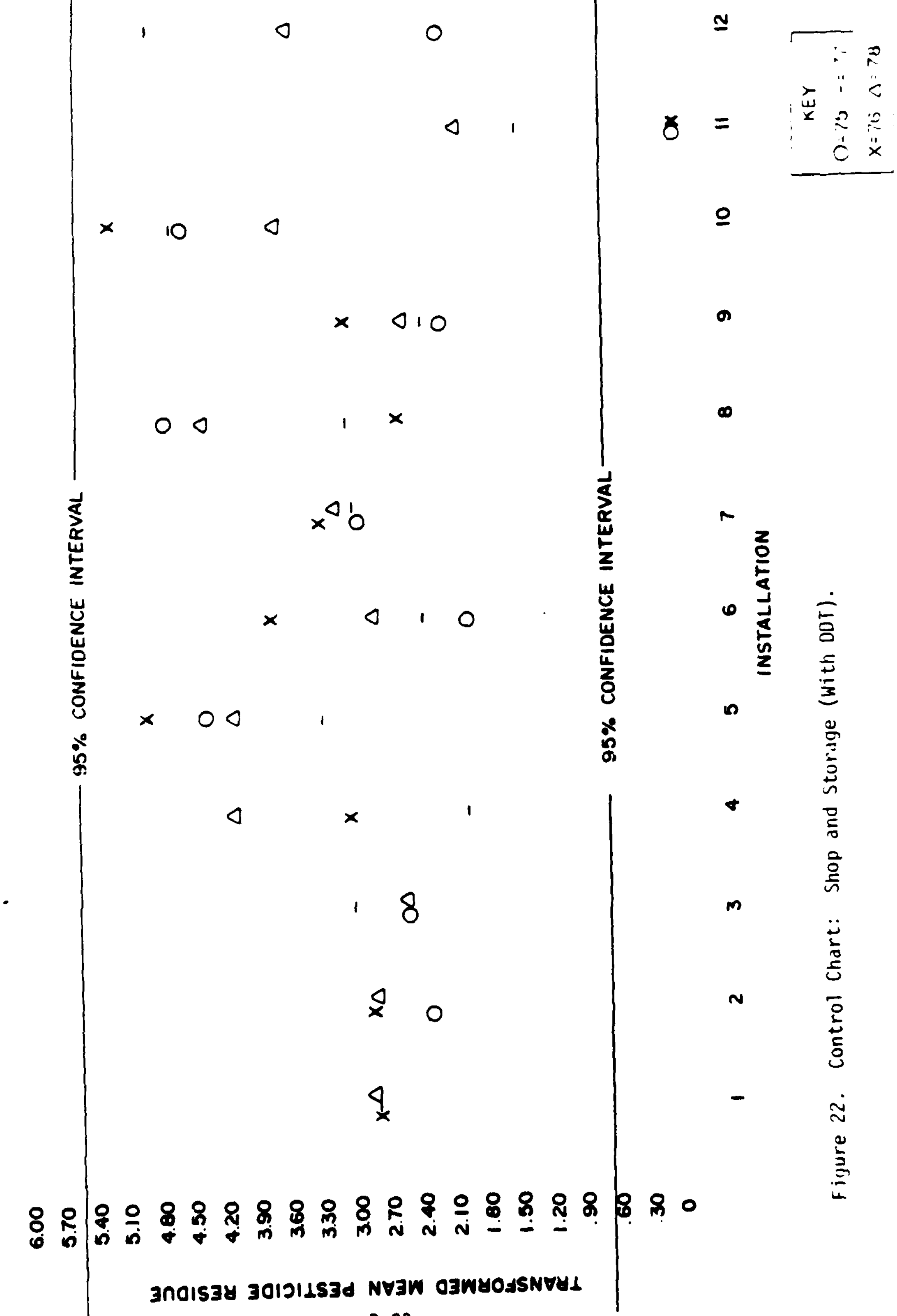


Figure 22. Control Chart: Shop and Storage (With DDT).

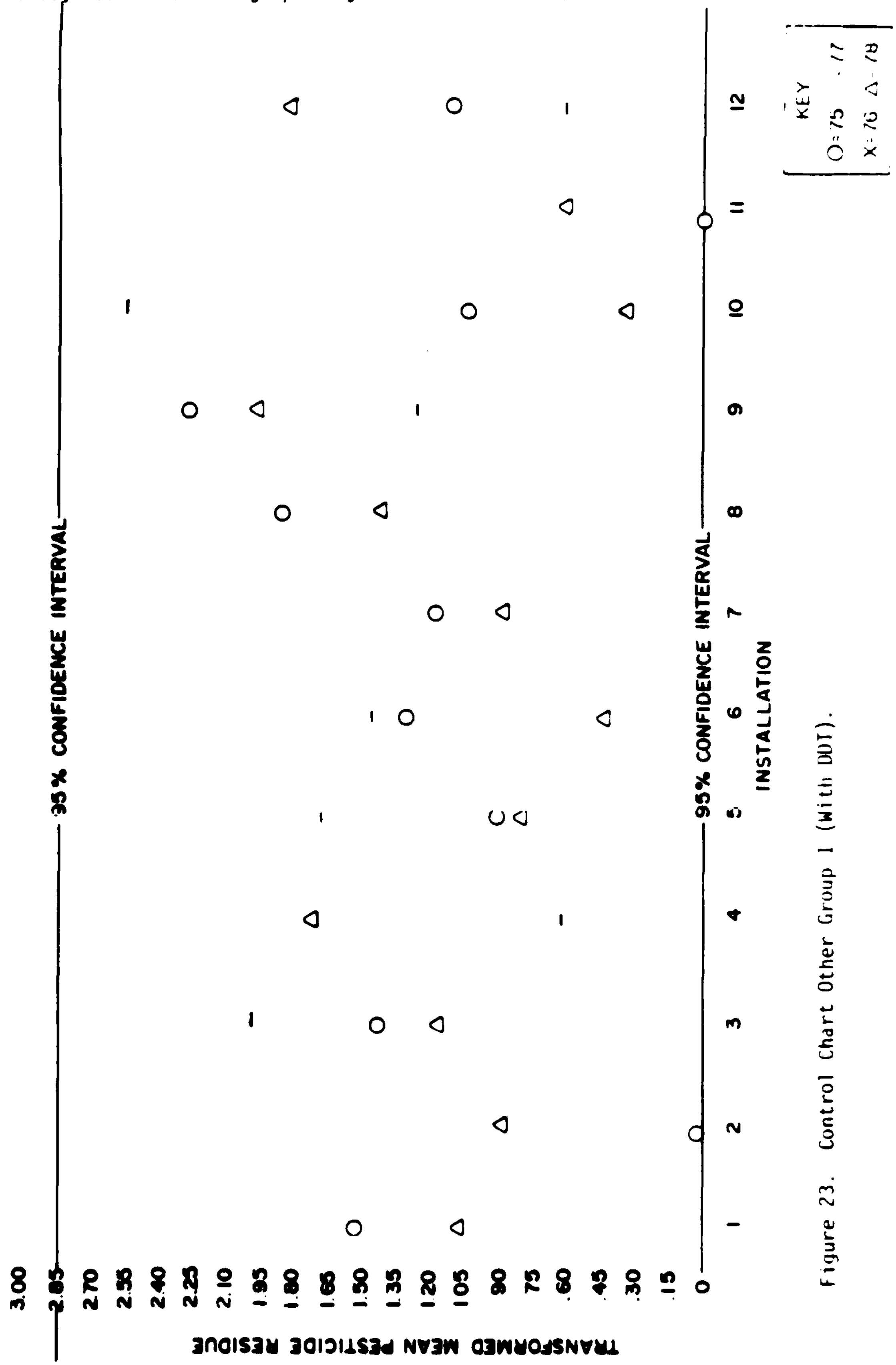


Figure 23. Control Chart Other Group I (With DDT).

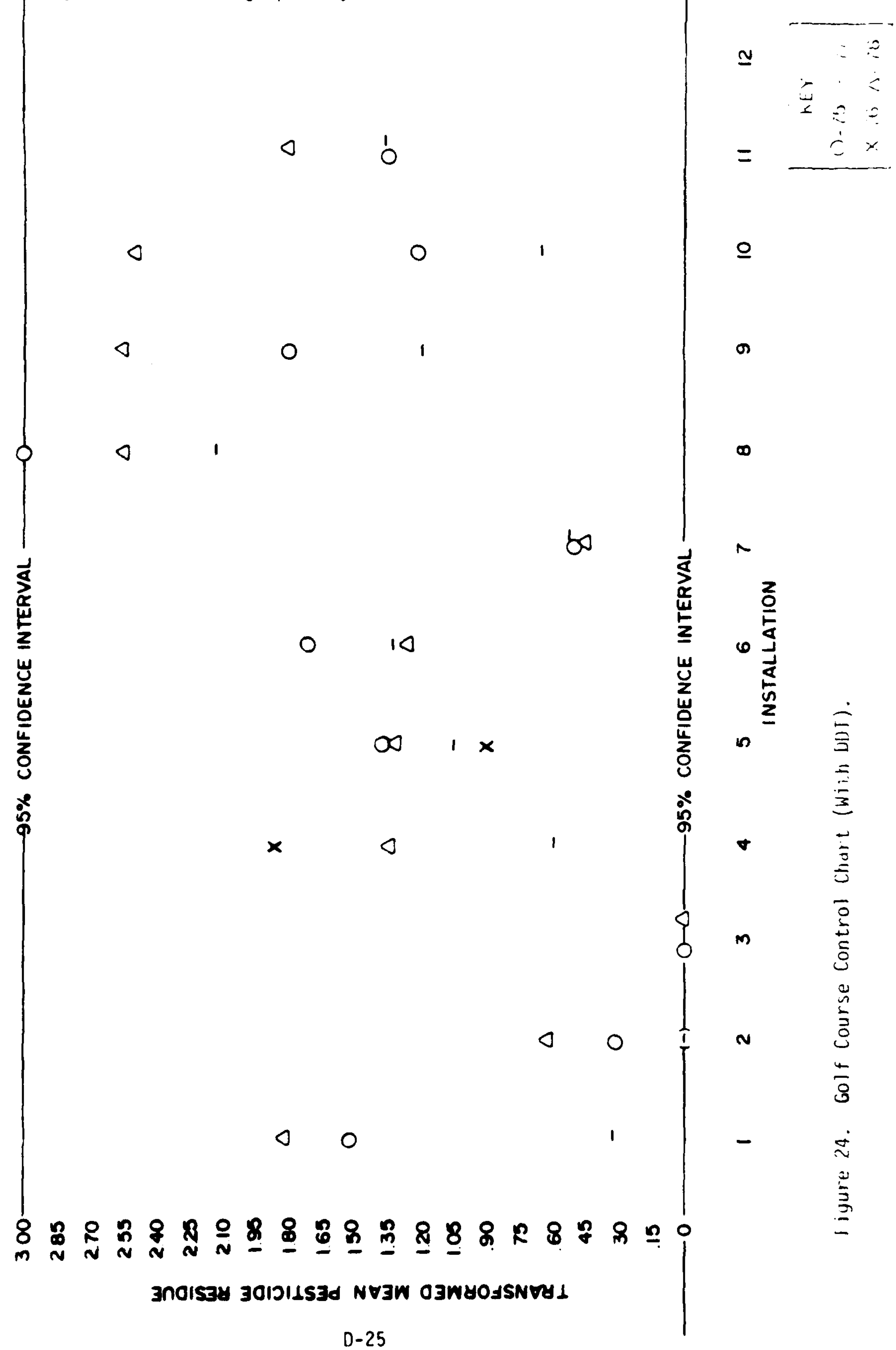


Figure 24. Golf Course Control Chart (With DDT).

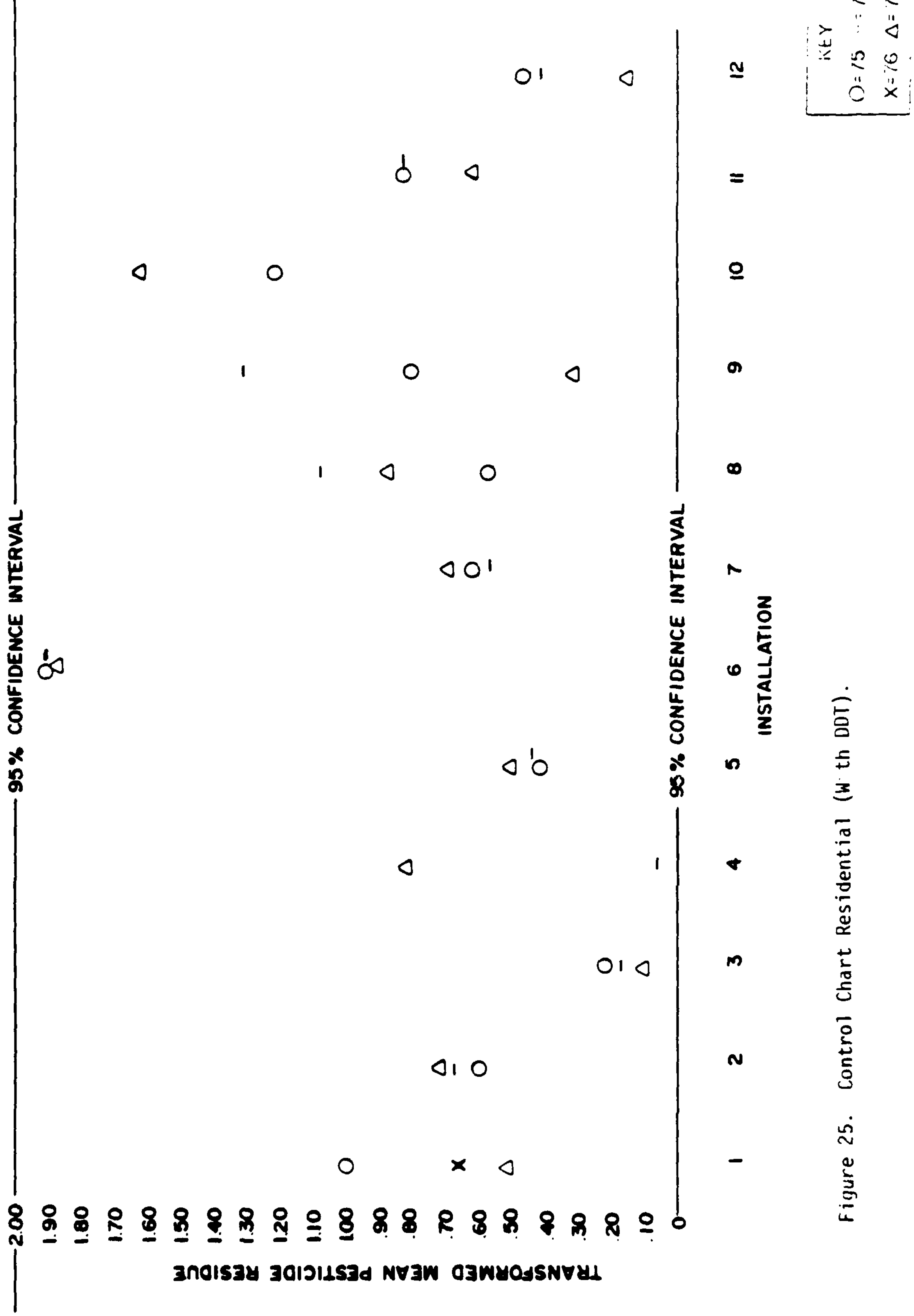
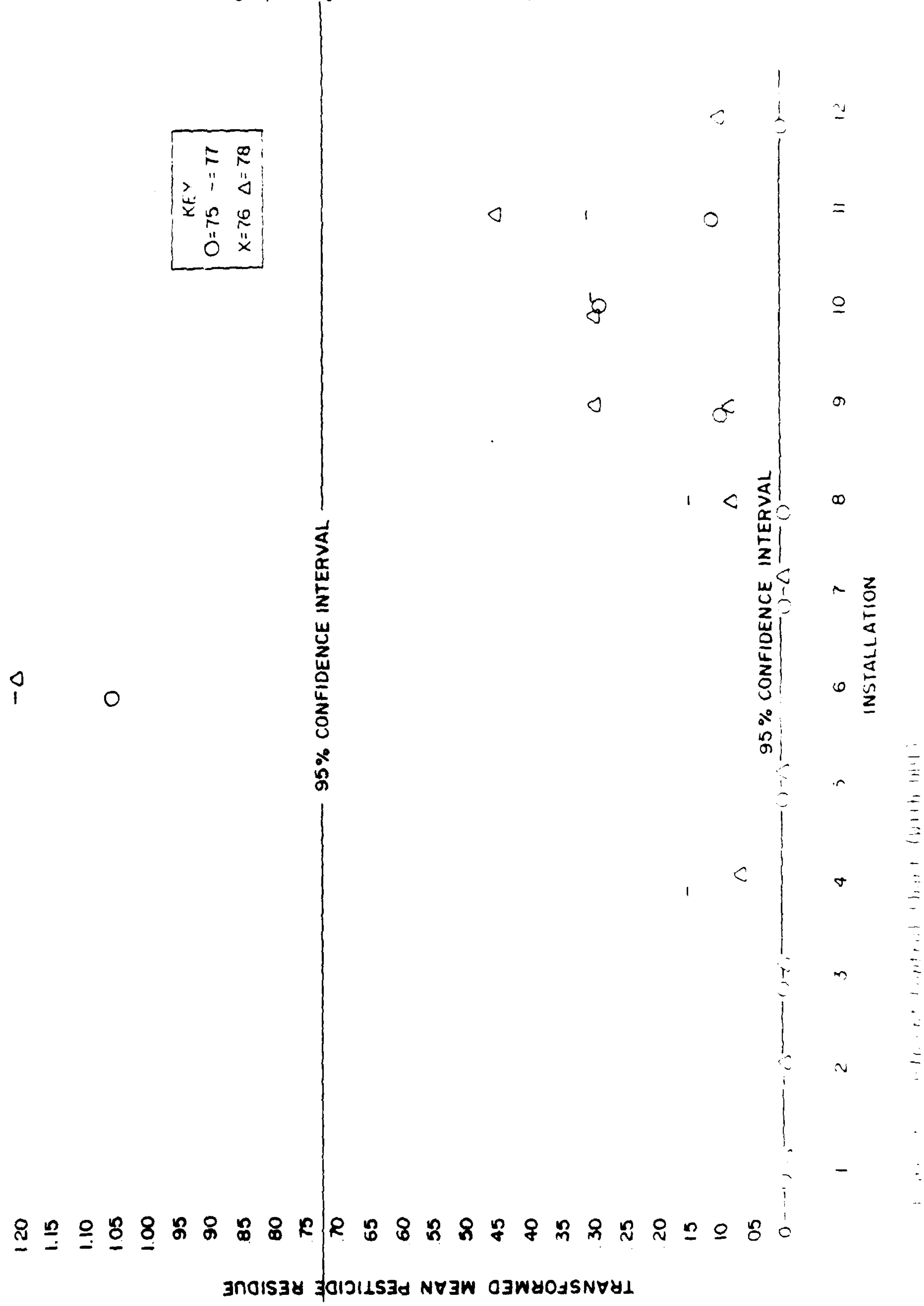


Figure 25. Control Chart Residential (W th DDT).


KEY
O=75 -=77
X=76 Δ=78
95% CONFIDENCE INTERVAL
95% CONFIDENCE INTERVAL
INSTALLATION
TRANSFORMED MEAN PESTICIDE RESIDUE

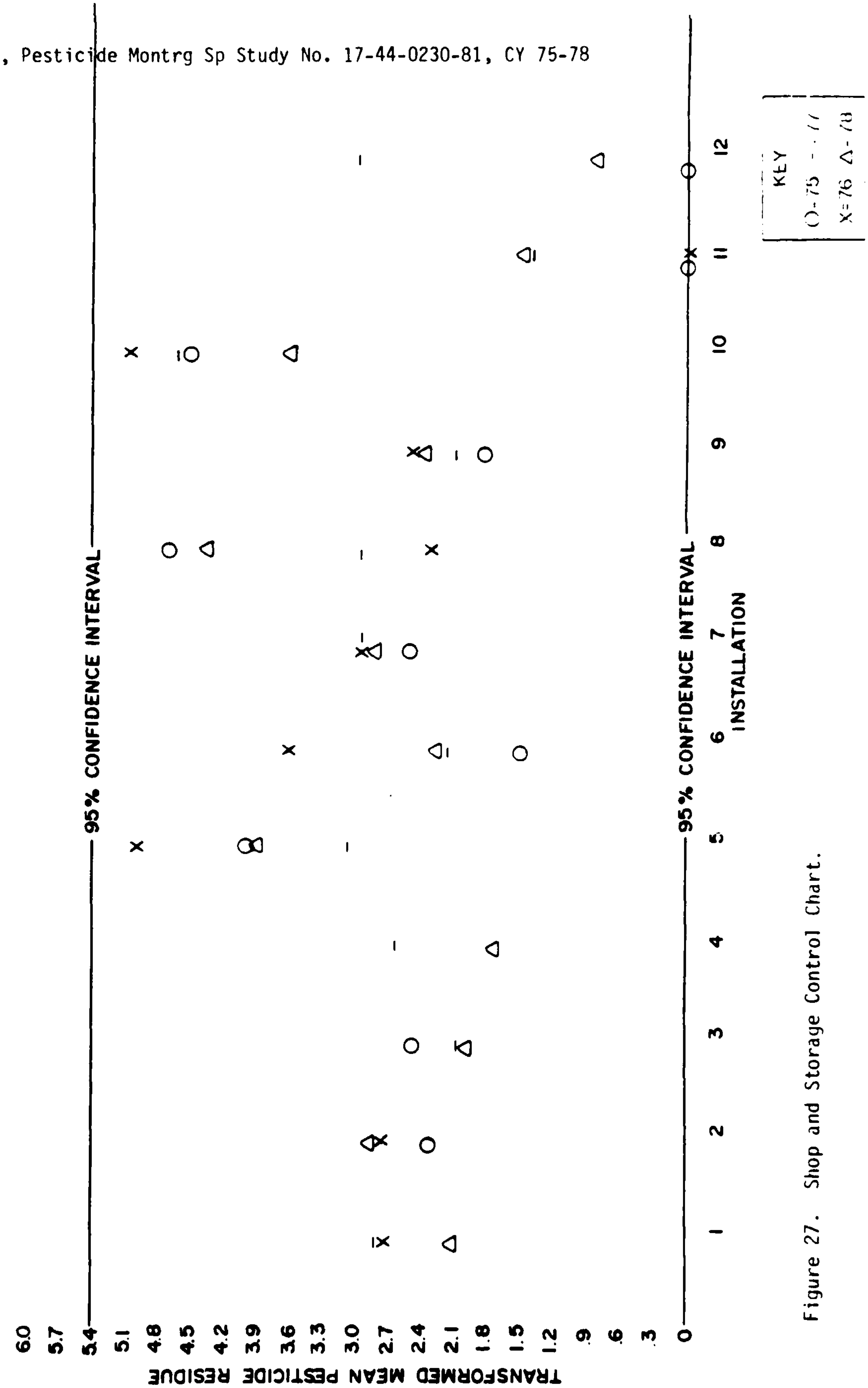


Figure 27. Shop and Storage Control Chart.

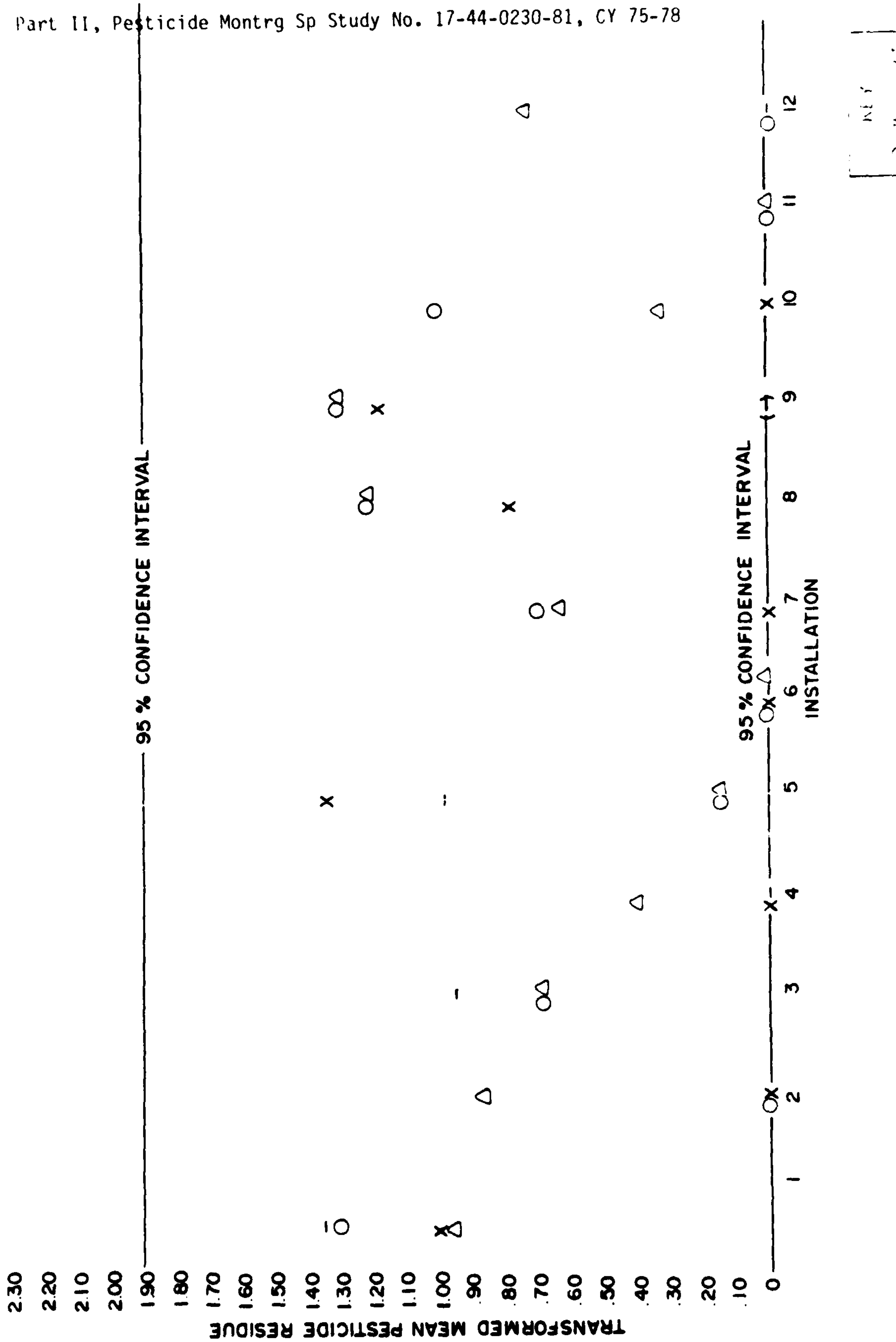


Figure 28. Other Group I Control Chart.

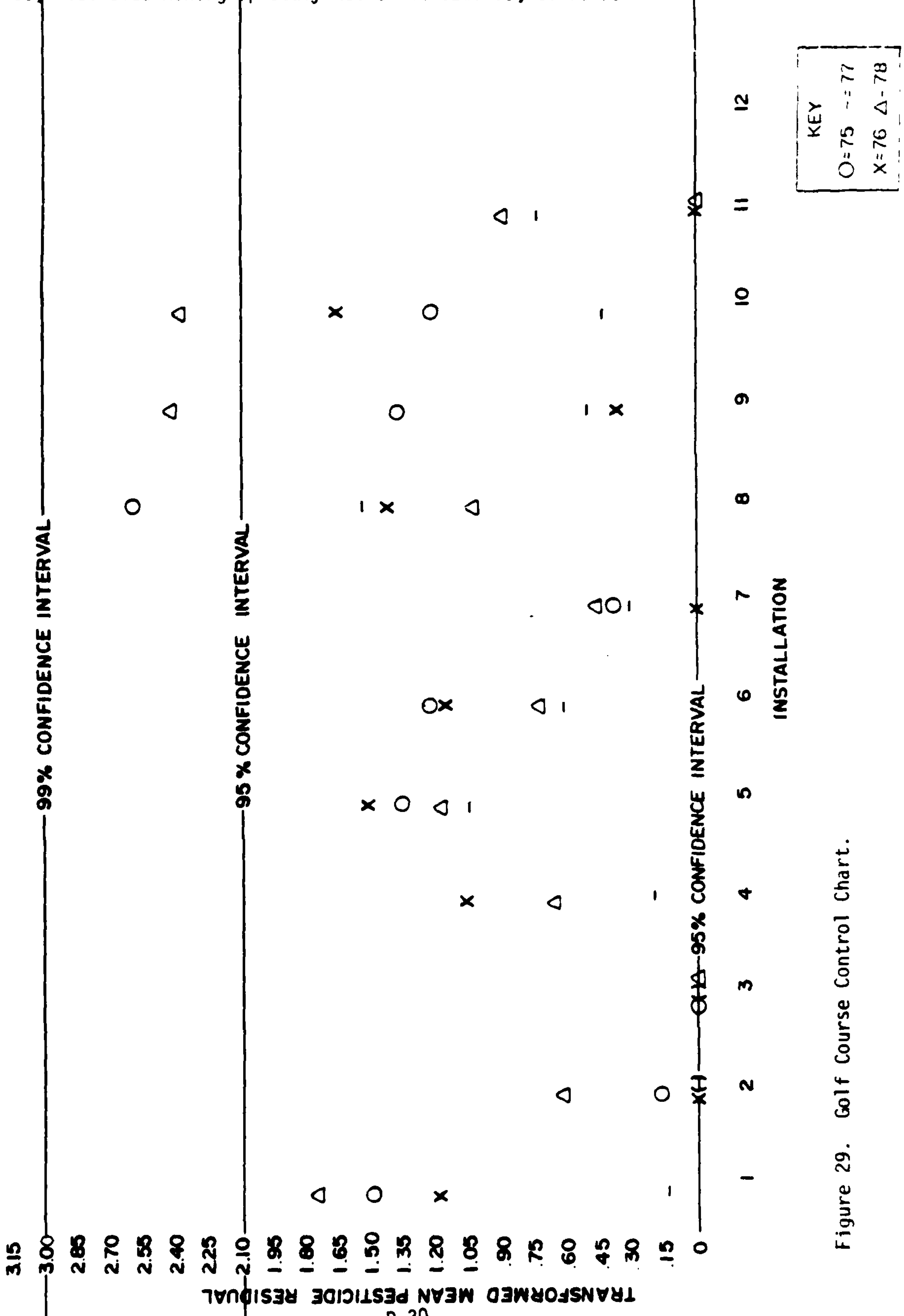


Figure 29. Golf Course Control Chart.

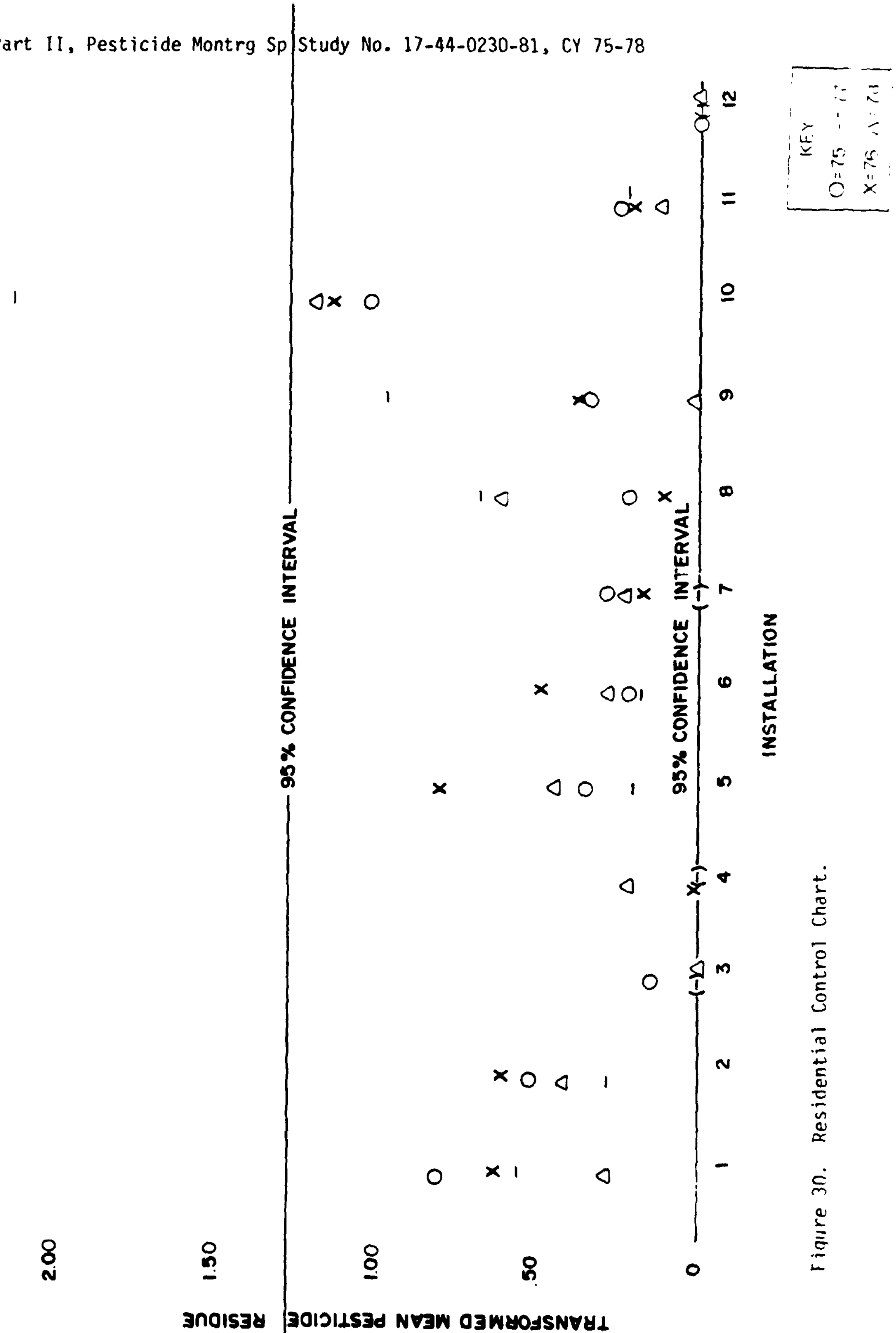


Figure 30. Residential Control Chart.

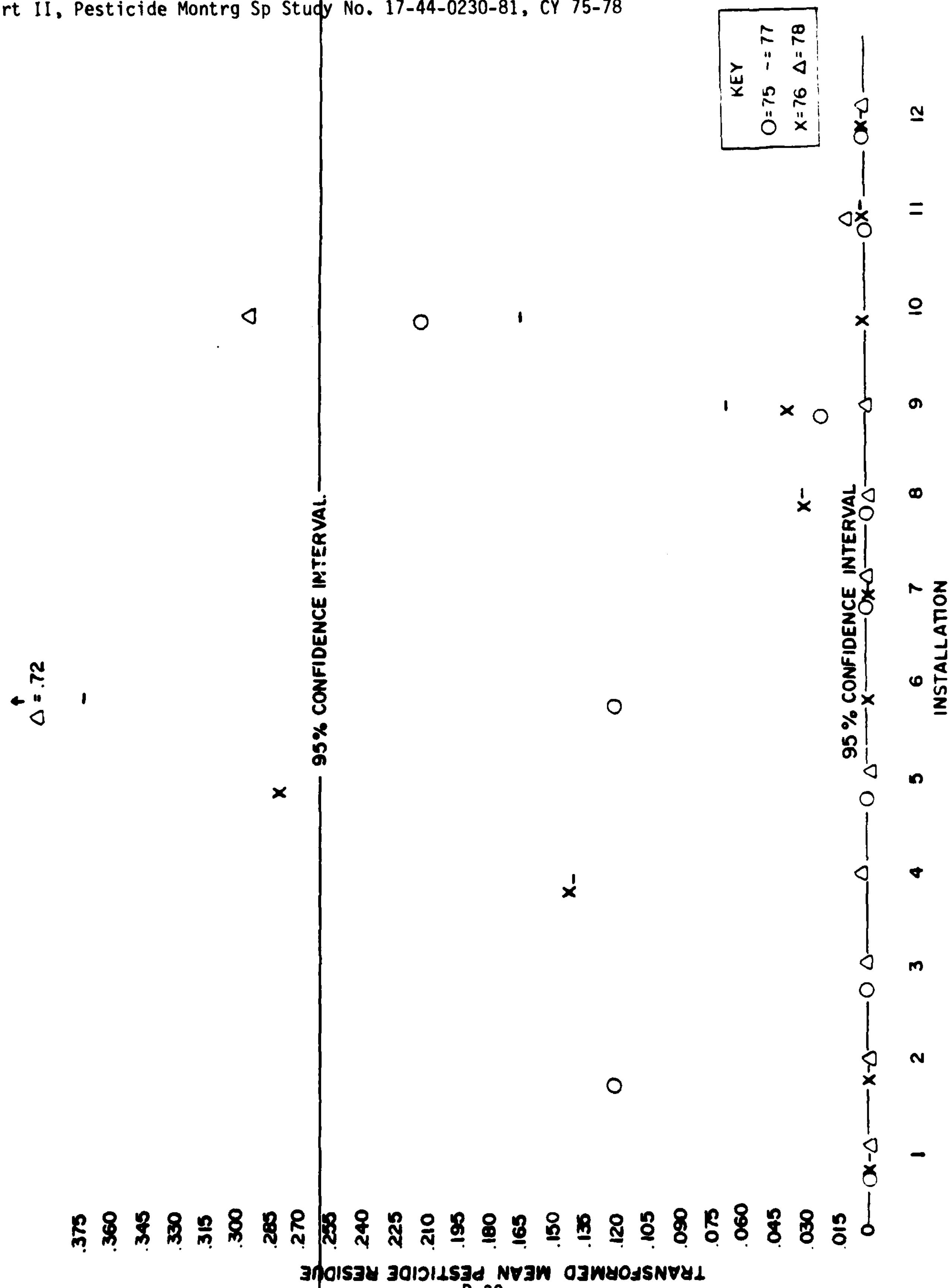


Figure 31. Sediment Control Chart.

APPENDIX E

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